



4th International Symposium on Growth of III-Nitrides

16-19 July, 2012, St. Petersburg, Russia



ABSTRACT BOOK

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July 16-19, 2012, St. Petersburg, Russia

Preface

Welcome to the *4th International Symposium on Growth of III-Nitrides* (ISGN4) which is organized in 2012 in Saint-Petersburg, Russia, by the Ioffe Physical Technical Institute of Russian Academy of Sciences, the world known research center in the field of semiconductor physics and technology.

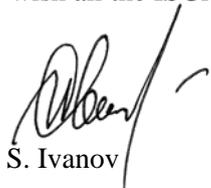
This is a rather new Symposium series started in 2006 (Linköping, Sweden) and continued in 2008 (Izu, Japan) and 2010 (Montpellier, France). It supplements two big biennial alternating III-Nitride meetings IWN and ICNS considering all the aspects of III-Nitride physics. ISGN4 is the main international topical symposium focusing directly on key issues of III-Nitride technology. It covers all aspects of growth and studies of bulk materials (AlN, GaN, InN) and their alloys, as well as fabrication of templates, epitaxial layers and low-dimensional structures, and also considers novel device concepts for optoelectronics and electronics. Practically all employed and newly developed growth techniques, novel characterization tools, and devices breakthroughs are discussed here.

In spite of the extremely fast development and commercial implementation of bright UV and visible LEDs/LDs, high efficiency white LED-lamps for general lighting, GaN-based high-power and high-temperature ultrahigh frequency HEMT transistors, which sometimes go ahead of understanding of fundamental physics and material science beneath, the potential of these materials is far from being exhausted. The most important problem is a quite narrow composition range of commercially employed compounds, extending from GaN by only 15-20 mol. % toward the Al- and In-rich sides. This is mainly caused by a big difference in lattice parameters and thermal properties between the constituent binary compounds, deficiency of low-cost high-quality homoepitaxial substrates (GaN and especially AlN), and difficulties in p-doping of high In- and Al-rich compounds. Recently, several important breakthroughs have been made in different fields of III-Nitride technology. In particular, development of various technologies of high-quality bulk GaN and AlN crystals suitable for fabrication of polar, semi- and non-polar epitaxial substrates resulted in demonstration of efficient blue-green LDs (520-540 nm), deep UV LEDs and low-threshold optically-pumped lasers in the 250-300 nm range. Recent progress in plasma-assisted molecular beam epitaxy has led to elaboration of AlGaN/GaN cubic low-dimensional structures, 3D nanostructured LEDs, novel type of quantum cascade emitters aimed at IR and THz spectral ranges. The technological advances are supported by deep fundamental studies of structural, optical and electrical properties, using sophisticated experimental tools. All these issues are presented in the ISGN4 Scientific Program.

The attendance of the Symposium is pretty high and world wide: approximately 250 scientists from 25 countries. Therefore the Symposium format has been increased from a three-day to four-day meeting. The Program is very intense and includes 1 plenary, 22 invited, and 48 oral talks, as well as two large poster sessions (~170 posters). The Symposium proceedings will be published in a special issue of *Physica Status Solidi* not later than early spring 2013. ISGN4 is accompanied by the Exhibition of novel technological equipment and materials presented by world leading and relatively young companies: Aixtron, Riber, Veeco, Cree, Camesa, Omicron, SemiTEq, and some others.

On behalf of the International Advisory Committee and Organizing Committee we would like to thank all the Official Partners and Sponsors of ISGN4 for their substantial support which has allowed many young scientists to participate in the meeting with a reduced fee as well as has given an opportunity for Organizers to support many regular participants.

We wish all the ISGN4 participants an enjoyable and successful meeting.



S. Ivanov



H. Amano



B. Gil

Saint-Petersburg, June 10, 2012

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CALENDAR OF EVENTS

Sunday, July 15

17:00 – 20:30 Registration

Monday, July 16

8:00 – 18:00 Registration

8:45 – 9:00 Opening remarks

9:00 - 9:45 Plenary paper

9:45 - 10:45 **Session 1** “Novel III-Nitride growth techniques”

10:45 - 11:15 Coffee-break

11:15 - 13:00 **Session 2** “AlGaN alloys for deep UV”

13:00 - 14:15 Lunch

14:15 - 16:00 **Session 3** “Growth of bulk crystals and templates”

16:00 - 16:30 Coffee-break

16:30 - 18:00 **Session 4** “Device quantum structures”

18:00 - 19:30 Poster session I

20:00 - 22:00 Welcome reception

Tuesday, July 17

8:00 – 18:00 Registration

9:00 - 10:45 **Session 5** “MBE growth of quantum structures”

10:45 - 11:15 Coffee-break

11:15 - 13:00 **Session 6** “Growth of GaInN QW LDs and solar cells”

13:00 - 14:15 Lunch

14:15 - 16:00 **Session 7** “Nanowires”

16:00 - 16:30 Coffee-break

16:30 - 18:00 **Session 8** “Magnetic and novel materials”

18:00 - 19:30 Poster session II

Wednesday, July 18

8:00 – 13:00 Registration

9:00 - 10:45 **Session 9** “Microstructure studies”

10:45 - 11:15 Coffee-break

11:15 - 13:00 **Session 10** “MOCVD growth of quantum structures”

13:00 - 18:00 Symposium Excursion

19:00 - 23:00 Symposium Dinner

Thursday, July 19

8:00 – 12:00	Registration
9:00 - 10:45	Session 11 “Optical properties”
10:45 - 11:15	Coffee-break
11:15 - 13:00	Session 12 “III-N Growth on Si”
13:00 - 14:15	Lunch
14:15 - 15:45	Session 13 “Defects and impurities”
15:45 - 16:15	Coffee-break
16:15 - 18:00	Session 14 “3D Nanostructures and Devices”
18:00 - 18:20	Closing remarks

ORAL SESSIONS

Monday, July 16

		page
Welcome greetings	8:45 - 9:00	
Plenary paper	9:00 - 9:45	
Chair: S. Ivanov		
Review on growth, properties, and applications of AlInN		48
<u>N. Grandjean</u> <i>Institute of Condensed Matter Physics, Ecole Polytechnique Fédérale de Lausanne (EPFL), Switzerland</i>		
Session 1 “Novel III-Nitride growth techniques”	9:45 - 10:45	
Chair: S. Chichibu		
Mo-1i	9:45 - 10:15	
Future Prospect of Large Area Nitride Devices Prepared by Pulsed Sputtering Deposition		49
<u>H. Fujioka</u> <i>Institute of Industrial Science, University of Tokyo, Japan; CREST, Japan Science and Technology Corporation, Japan</i>		
Mo-2i	10:15 - 10:45	
Semipolar nitride hetero-structures on patterned substrates		50
<u>F. Scholz</u> , T. Meisch, R. Leute, I. Argut, S. Schwaiger, I. Tischer, K. Thonke, S. Metzner, F. Bertram, J. Christen, H. Lengner, J. Thalmer, and J. Zweck <i>Institute of Optoelectronics, University of Ulm, Germany; Institute of Quantum Matter, University of Ulm, Germany; Inst. of Exp. Physics, Otto-von-Guericke-University, Germany; Inst. of Exp. and Appl. Physics, Univ. of Regensburg, Germany; Osram AG, Germany</i>		
Coffee Break	10:45 - 11:15	
Session 2 “AlGaN alloys for deep UV”	11:15 - 13:00	
Chair: H. Amano		
Mo-3i	11:15 - 11:45	
AlN-based technology for deep UV and high-power applications		51
<u>Z. Sitar</u> , B. Moody, S. Craft, R. Schlessler, R. Dalmau, J. Xie, S. Mita, T. Rice, J. Tweedy, J. LeBeau, L. Hussey, R. Collazo, B. Gaddy, D. Irving <i>HexaTech, Inc., USA; Department of Materials Science and Engineering, North Carolina State University, USA</i>		
Mo-4i	11:45 - 12:15	
UV-C LEDs on free-standing AlN substrates		52
<u>R.T. Bondokov</u> , S.P. Rao, S.R. Gibb, J.R. Grandusky, J. Chen and L.J. Schowalter <i>Crystal IS, Inc. USA</i>		

		page
Mo-1o	12:15 - 12:30 Efficient current injection in 290 nm to 350 nm UV light emitting diodes <u>F. Mehnke</u> , J. Stellmach, T. Kolbe, M.-A. Rothe, C. Reich, T. Wernicke, M. Pristovsek, and M. Kneissl <i>Institute of Solid State Physics, Technische Universität Berlin, Germany</i>	53
Mo-2o	12:30 - 12:45 Improvement of light extraction efficiency of AlGaIn deep-UV LED using 2-dimensional photonic crystal (2D-PhC) <u>S. Fujikawa</u> , H. Hirayama, Y. Kashima, H. Nishihara, T. Tashiro, T. Ohkawa, S. W. Youn and H. Takagi <i>RIKEN, Japan; JST-CREST, Japan; Marubun Co. Ltd., Japan; Toshiba machine Co., Ltd., Japan; AIST, Japan</i>	54
Mo-3o	12:45 - 13:00 AlGaIn MSM photodetectors for the UV-C spectral region on planar and ELO AlN/sapphire templates A. Knigge, M. Brendel, F. Brunner, S. Einfeldt, V. Küller, <u>M. Weyers</u> <i>Ferdinand-Braun-Institut, Leibniz-Institut für Höchstfrequenztechnik, Germany</i>	55
<u>Lunch</u>		13:00 - 14:15
Session 3 “Growth of bulk crystals and templates” Chair: I. Grzegory		14:15 - 16:00
Mo-5i	14:15 - 14:45 Growth of bulk GaN by ammonothermal technique <u>R. Dwiliński</u> , R. Doradziński, R. Kucharski, M. Zając <i>AMMONO S.A., Poland</i>	56
Mo-4o	14:45 - 15:00 Specific features of sublimation growth of bulk AlN crystals on SiC wafers <u>E. Mokhov</u> , I. Izmaylova, O. Kazarova, A. Wolfson, S. Nagalyuk, D. Litvin, A. Vasiliev, H. Helava, Yu. Makarov <i>Ioffe Physical Technical Institute, Russia; Nitride Crystals Ltd., Russia; Nitride Crystals Inc., USA</i>	57
Mo-5o	15:00 - 15:15 Growth of cm-order bulk GaN single crystals by Na-flux method with a necking technique <u>M. Imade</u> , K. Murakami, H. Imabayashi, H. Takazawa, Y. Todoroki, D. Matsuo, A. Kitamoto, M. Maruyama, M. Yoshimura, and Y. Mori <i>Division of Electric, Electronic and Information Engineering, Osaka University, Japan</i>	58
Mo-6o	15:15 - 15:30 Crystal Growth of AlN Single Crystal by Sublimation method <u>S. Nagata</u> , H. Akiyama <i>JFE MINERAL COMPANY, LTD., Research Laboratories, Japan</i>	59

		page
Mo-6i	15:30 - 16:00 HVPE grown GaN: conducting and insulating substrates of various polarities <u>T. Paskova</u> <i>North Carolina State University, USA</i>	60
	<u>Coffee Break</u>	16:00 - 16:30
	Session 4 “Device quantum structures” Chair: A. Toropov	16:30 - 18:00
Mo-7i	16:30 - 17:00 GaN Quantum Devices for Infrared Optoelectronics <u>E. Monroy</u> , Y. Kotsar, A. Das, S. Valdueza-Felip, and E. Bellet-Amalric, C. Bougerol and R. Songmuang, L. Rapenne and E. Sarigiannidou, S. Sakr, M. Tchernycheva, and F. H. Julien, E. Gross, A. Pesach, and G. Bahir <i>CEA-CNRS Group “Nanophysique et semiconducteurs”, CEA-INAC-SP2M, France; CEA-CNRS Group “Nanophysique et Semiconducteurs”, Institut Néel-CNRS, France; INP-Grenoble/Minatec, France; Institut d'Electronique Fondamentale-CNRS, Univ. Paris-Sud 11, France; Technion-Israel Institute of Technology, Israel</i>	61
Mo-7o	17:00 - 17:15 Intersubband transitions at atmospheric window in Al_xGa_{1-x}N/GaN multiple quantum wells grown on GaN/sapphire templates adopting AlN/GaN superlattices interlayer C.C. Huang, <u>B. Shen</u> , F. J. Xu, Z. Y. Xu, L. B. Cen, X. Q. Wang, Z. J. Yang <i>State Key Laboratory of Artificial Microstructure and Mesoscopic Physics, School of Physics, Peking University, China</i>	62
Mo-8o	17:15 - 17:30 Growth by ammonia source Molecular Beam Epitaxy of an AlGaIn/GaN HEMT with an InGaIn back-barrier <u>S. Rennesson</u> , B. Damilano, P. Vennéguès, S. Chenot and Y. Cordier <i>CRHEA-CNRS, France; Physics Department, University of Nice-Sophia Antipolis, France</i>	63
Mo-9o	17:30 - 17:45 Regrown Source/Drain for Enhancement-Mode AlN/GaN MOSHFETs on Si substrate Exhibiting High Current and Transconductance T. Huang, X. Zhu, and <u>K.M. Lau</u> <i>Department of Electronic and Computer Engineering, Hong Kong University of Science & Technology, Hong Kong</i>	64

	page
Mo-10o 17:45 - 18:00 Growth Optimization of AlGaN/AlN/GaN HEMT Epitaxial Structures by MOCVD A. Arendarenko, I. Ermoshin, V. Oreshkin, <u>Yu. Sveshnikov</u> and I. Tsypfenkov <i>JSC Elma-Malachit, Russia</i>	65
<u>Poster session I</u> 18:00 - 19:30	22
<u>Welcome reception</u> 20:00 - 22:00	

Tuesday, July 17

Session 5 “MBE growth of quantum structures” 9:00 - 10:45 Chair: Y. Nanishi	
Tu-1i 9:00 - 9:30 Kinetics of metal-rich PA MBE of AlGaN-heterostructures for UV-optoelectronics <u>V.N. Jmerik</u> <i>Ioffe Physical Technical Institute, Russia</i>	66
Tu-2i 9:30 - 10:00 MBE growth and applications of cubic AlN/GaN quantum wells <u>D.J. As</u> <i>University of Paderborn, Department of Physics, Germany</i>	67
Tu-1o 10:00 - 10:15 GaN/(Al,Ga)N heterostructures grown on (Zn,Mg)O by ammonia source molecular beam epitaxy <u>Y. Xia</u> , J.Brault, P. Vennéguès, M. Nemoz, M. Teisseire, M. Leroux, J.-M. Chauveau <i>CRHEA-CNRS, France; University of Nice Sophia-Antipolis, France</i>	68
Tu-2o 10:15 - 10:30 Molecular Beam Epitaxy of High Electron Mobility InN and High Quality In_xGa_{1-x}N Layers (0≤x≤1) <u>X.Q. Wang</u> , S.T. Liu, G. Chen and B. Shen <i>State Key Laboratory of Artificial Microstructure and Mesoscopic Physics, School of Physics, Peking University, China</i>	69
Tu-3o 10:30 - 10:45 In-situ monitoring of InGaN growth using DERI method <u>T. Araki</u> , N. Uematsu, M. Yutani, T. Saito, J. Sakaguchi, T. Yamaguchi, T. Fujishima, E. Matioli, T. Palacios, Y. Nanishi <i>Ritsumeikan Univ., Dept. of Photonics, Kusatsu, Japan; Kogakuin Univ., Japan; Massachusetts Institute of Technology, USA; Ritsumeikan Univ., Japan; Seoul National Univ., WCU Program, Korea</i>	70
<u>Coffee Break</u> 10:45 - 11:15	

		page
Session 6 “Growth of GaInN QW LDs and solar cells”	11:15 - 13:00	
	Chair: N. Grandjean	
Tu-3i	11:15 - 11:45 True green LDs on c-plane: growth and LD parameters <u>A. Avramescu</u> , T. Lermer, C. Eichler, G. Bruederl, T. Hager, S. Gerhard, U. Strauss <i>OSRAM Opto Semiconductors GmbH, Germany</i>	71
Tu-4i	11:45 - 12:15 Development of semipolar GaN-based laser diodes <u>D. Sizov</u> , R. Bhat, C. Zah <i>Corning Incorporated, USA</i>	72
Tu-4o	12:15 - 12:30 GaN substrates grown by HNPS method in Multi-Feed-Seed configuration and their application for laser diode arrays with variable wavelength <u>I. Grzegory</u> , M. Sarzyński, M. Boćkowski, T. Suski, R. Czernecki, P. Perlin, and M. Leszczyński <i>Institute of High Pressure Physics PAS, Poland; TopGaN Ltd, Poland</i>	73
Tu-5o	12:30 - 12:45 Structure and stability of InGaN-based active regions in laser diodes grown by MOVPE <u>A. Mogilatenko</u> , V. Hoffmann, C. Netzel, U. Zeimer, S. Einfeldt, M. Weyers, M. Kneissl, G. Tränkle <i>Ferdinand-Braun-Institut, Leibniz-Institut fuer Hoechstfrequenztechnik, Germany; Institut für Festkoerperphysik, Technische Universitaet Berlin, Germany</i>	74
Tu-6o	12:45 - 13:00 InGaN prepared by pulsed sputtering and its application to solar cells <u>K. Morita</u> , M. Katoh, J. Ohta, S. Inoue, H. Fujioka <i>Institute of Industrial Science, Univ. of Tokyo, Japan; Core Research for Evolutional Science and Technology (CREST), Japan Science and Technology Agency (JST), Japan</i>	75
<u>Lunch</u>	13:00 - 14:15	
Session 7 “Nanowires”	14:15 - 16:00	
	Chair: A. Waag	
Tu-5i	14:15 - 14:45 III-N nanocolumn growth, properties and LEDs <u>K. Kishino</u> , V. Ramesh, K. Yamano, and S. Ishizawa <i>Sophia University, Japan; Sophia Nanotechnology Research Center, Sophia University, Japan</i>	76

		page
Tu-7o	14:45 - 15:00 Photodetection properties of single GaN nanowires <u>F. González-Posada</u> and E. Monroy, R. Songmuang and M. Den Hertog <i>CEA-CNRS Group “Nanophysique et semiconducteurs”, INAC-SP2M, CEA- Grenoble, France; CEA-CNRS Group “Nanophysique et Semiconducteurs”, Institut Néel-CNRS, France</i>	77
Tu-8o	15:00 - 15:15 Semi-polar GaN quantum wells grown on {11-2n} planes at the pyramidal tip of GaN nanocolumn arrays A. Urban, C. Oppo, J. Malindretos, <u>A. Rizzi</u> , E. Secco, N. Garro, A. Cantarero <i>IV. Physikalisches Institut, Georg-August-Universität Göttingen, Germany; Institut de Ciència dels Materials, Universitat de València, Spain</i>	78
Tu-9o	15:15 - 15:30 The control of crystal polarity for MOCVD nanowire growth <u>C. Durand</u> , X. J. Chen, D. Salomon, C. Bougerol, D. Sam-Giao, G. Perillat-Merceroz, B. Gayral and J. Eymery <i>Equipe mixte CEA-CNRS “Nanophysique et semiconducteurs”, SP2M, UMR-E CEA/UJF-Grenoble 1, INAC, France; CEA-Leti, MINATEC campus, France; Equipe mixte CEA-CNRS “Nanophysique et semiconducteurs”, Institut Néel, France; LEMMA, SP2M, UMR-E CEA/UJF-Grenoble 1, INAC, France</i>	79
Tu-10o	15:30 - 15:45 Scaling growth laws of self-induced GaN nanowires on Si substrates <u>V.G. Dubrovskii</u> , A.D. Bolshakov, V. Consonni, A. Trampert, L. Geelhaar, and H. Riechert <i>St. Petersburg Academic University, Russia; Ioffe Physical Technical Institute, Russia; Laboratoire des Matériaux et du Génie Physique, CNRS – Grenoble INP, France; Paul-Drude-Institut für Festkörperelektronik, Germany</i>	80
Tu-11o	15:45 - 16:00 Localized surface plasmons enhancement of GaN/InGaN LED performance: problems and possible solutions <u>A.Y. Polyakov</u> , Lee-Woon Jang, Dae-Woo Jeon, and In-Hwan Lee <i>School of Advanced Materials Engineering and Research Center for Advanced Materials Development, Chonbuk National University, Korea; Institute of Rare Metals, Russia</i>	81

Coffee Break

16:00 - 16:30

		page
Session 8 “Magnetic and novel materials”	16:30 - 18:00	
	Chair: B. Gil	
Tu-6i	16:30 - 17:00 Controlling (ferro)magnetism in III-Nitrides <u>A. Bonanni</u> <i>Institut für Halbleiter- und Festkörperphysik, Johannes Kepler University, Austria</i>	82
Tu-12o	17:00 - 17:15 Theory of transition metal aggregation and spin-spin interactions in III-nitrides N. Gonzalez Szwacki, <u>J. A. Majewski</u> , and T. Dietl <i>Institute of Physics, Polish Academy of Sciences, Poland; Institute of Theoretical Physics, Faculty of Physics, University of Warsaw, Poland</i>	83
Tu-13o	17:15 - 17:30 Scandium-based nitrides: new members of the III-nitride family <u>M.A. Moram</u> , S.M. Knoll, H.E. Beere, S. Zhang, M.J. Kappers, C.J. Humphreys, D.A. Ritchie <i>Dept. Materials, Imperial College London, UK; Dept. Physics, University of Cambridge, UK; Dept. Materials Science, University of Cambridge, UK</i>	84
Tu-14o	17:30 - 17:45 Molecular beam epitaxy of highly mismatched N-rich GaN_{1-x}Sb_x alloys <u>S. V. Novikov</u> , K. M. Yu, A. Levander, D. Detert, W. L. Sarney, R.W. Martin, O.D. Dubon, J. Wu, S. P. Svensson, W. Walukiewicz, and C. T. Foxon <i>School of Physics and Astronomy, University of Nottingham, UK; Materials Sciences Division, Lawrence Berkeley National Laboratory, USA; Department of Materials Science & Engineering, University of California, USA; US Army Research Laboratory, USA; Department of Physics, SUPA, University of Strathclyde, UK</i>	85
Tu-15o	17:45 - 18:00 In-situ RF-MBE growth of AlO_x/n-GaN composite structures <u>M. Higashiwaki</u> , T. Igaki, T. Yamaguchi, and T. Honda <i>National Institute of Information and Communications Technology, Japan; PRESTO, Japan Science and Technology Agency, Japan; Department of Electrical Engineering and Electronics, Kogakuin University, Japan</i>	86
<u>Poster session II</u>	18:00 - 19:30	33

Wednesday, July 18

		page
Session 9 “Microstructure studies”	9:00 - 10:45	
Chair: J. Han		
We-1i	9:00 - 9:30 Nano-scale correlation of structural and optical properties in Group-III-Nitrides by (S)TEM-cathodoluminescence spectroscopy <u>J. Christen</u> <i>Otto-von-Guericke-University Magdeburg, Germany</i>	87
We-1o	9:30 - 9:45 EPR and ODMR defect control in AlN bulk crystals I.V.Ilyin, V.A. Soltamov, A.S. Gurin, D.O. Tolmachev, N.G. Romanov, E.N. Mokhov and <u>P.G. Baranov</u> <i>Ioffe Physical Technical Institute, Russia</i>	89
We-2o	9:45 - 10:00 Atom Probe Tomography Characterization of InGaN Device Structures <u>P.H. Clifton</u> , T.J. Prosa, D. Olson and D.J. Larson <i>Cameca Instruments Inc., USA</i>	90
We-3o	10:00 - 10:15 Observation of GaInN strain relaxation by <i>in situ</i> X-ray diffraction monitoring during metalorganic vapor phase epitaxy growth <u>M. Iwaya</u> , D. Iida, T. Sugiyama, M. Sowa, Y. Kondo, H. Matsubara, T. Takeuchi, S. Kamiyama, and I. Akasaki <i>Faculty of Science and Technology, Meijo University Japan; Akasaki Research Center, Nagoya University, Japan</i>	91
We-4o	10:15 - 10:30 EXAFS study of GaN/AlN multiple quantum wells grown by ammonia MBE <u>K. Zhuravlev</u> , I. Alexandrov, T. Malin, S. Trubina, and S. Erenburg, L. Dobos, and B. Pecz, V. Davydov, A. Smirnov, and R. Kyutt <i>A.V.Rzhanov Institute of Semiconductor Physics, Siberian Branch of the Russian Academy of Sciences, Russia; Nikolaev Institute of Inorganic Chemistry, Siberian Branch of the Russian Academy of Sciences, Russia; Institute for Technical Physics and Materials Science, Hungarian Academy of Sciences, Hungary; Ioffe Physical Technical Institute, of the Russian Academy of Sciences, Russia</i>	92

		page
We-5o	10:30 - 10:45 Characterisation of Nitride Materials by Synchrotron X-ray Microdiffraction Reciprocal Space Mapping <u>V. Kachkanov</u> , I.P. Dolbnya, K.P. O'Donnell, K. Lorenz, S. Pereira, R.W. Martin, P.R. Edwards, I.M. Watson <i>Diamond Light Source Ltd, United Kingdom; Department of Physics, SUPA, University of Strathclyde, United Kingdom; Instituto Tecnológico e Nuclear, Sacavem, Portugal; CICECO, Departamento de Física and I3N, Universidade de Aveiro, Portugal; Institute of Photonics, SUPA, University of Strathclyde, United Kingdom</i>	93
Coffee Break		10:45 - 11:15
Session 10 “MOCVD growth of quantum structures” Chair: F. Scholz		11:15 - 13:00
We-2i	11:15 - 11:45 MOVPE GaN on Si for Solid-State Lighting Applications D. Zhu, M.J. Kappers, E.J. Thrush, P. Dawson and <u>C.J. Humphreys</u> <i>Department of Materials Science and Metallurgy, University of Cambridge, UK; Photon Science Institute, School of Physics and Astronomy, University of Manchester, UK</i>	94
We-3i	11:45 - 12:15 Stimulated Formation of InGaN Quantum Dots by MOCVD <u>A. Tsatsulnikov</u> , W. Lundin, A. Nikolaev, A. Sakharov, N. Cherkashin and M. Hytch <i>Ioffe Physical Technical Institute, Russia; Center for Material Elaboration & Structural Studies (CEMES), National Center for Scientific Research (CNRS), France</i>	95
We-6o	12:15 - 12:30 Reactants injector temperature effect on III-Nitrides materials deposition in the high speed vertical rotating disc MOCVD reactor <u>A. Gurary</u> , B. Mitrovic, E. Armour, M. Pophristic, A. Paranjpe <i>Veeco Instruments Inc., USA</i>	96
We-7o	12:30 - 12:45 High-quality III-nitride templates with overgrown porous structure <u>M. Ali</u> , D.M. Artemiev, V.E. Bougrov, A.R. Kovsh, M.A. Odnoblyudov, and A.E. Romanov <i>Optogan GmbH, Germany; Optogan Group, Russia; National Research University ITMO, Russia; Ioffe Physical-Technical Institute RAS, Russia</i>	97

	page
We-8o 12:45 - 13:00 Three zone topside temperature control in a close coupled showerhead[®] MOVPE reactor <u>F. Schulte</u> , A. Boyd, O. Feron, P. Lauffer, X Chen, M Luenenbuerger, R Leiers, S Thomas, B Schineller, L. Pauli, J Lindner, M Heuken <i>AIXTRON SE, Germany</i>	98

Symposium Excursion 13:00 - 18:00

Symposium Dinner 19:00 - 23:00

Thursday, July 19

Session 11 “Optical properties” Chair: B. Monemar	9:00 - 10:45	
Th-1i 9:00 - 9:30 Time-resolved luminescence studies on AlN and high AlN mole fraction AlGaN alloys <u>S. F. Chichibu</u> and A. Uedono <i>Institute of Multidisciplinary Research for Advanced Materials, Tohoku University, Japan; Institute of Applied Physics, University of Tsukuba, Japan</i>	99	
Th-1o 9:30 - 9:45 Higher than 90% internal quantum efficiency of photoluminescence in GaN:Zn,Si <u>M. A. Reshchikov</u> , A. Behrends, A. Bakin, and A. Waag <i>Physics Department, Virginia Commonwealth University, USA; Institute of Semiconductor Technology, Technische Universität Braunschweig, Germany</i>	100	
Th-2o 9:45 - 10:00 Surface acoustic waves and elastic constants of InN epilayers determined by Brillouin scattering <u>R. J. Jiménez Riobóo</u> , N. Domènech-Amador, R. Cuscó, C. Prieto, T. Yamaguchi, Y. Nanishi and L. Artús <i>Instituto de Ciencia de Materiales de Madrid, CSIC, Spain; Institut Jaume Almera, CSIC, Spain; Faculty of Science and Engineering, Ritsumeikan University, Japan</i>	101	
Th-3o 10:00 - 10:15 Splitting of surface-related phonon modes in Raman spectra of self-assembled GaN nanowires J. Wang, <u>F. Demangeot</u> , R. Péchou, A. Ponchet, A. Cros and B. Daudin <i>CNRS-CEMES, France; Institute of Materials Science, Universidad de Valencia, Spain; CEA-CNRS Group, France; Université de Toulouse, UPS, France</i>	102	

		page
Th-4o	10:15 - 10:30 Linear and nonlinear optical investigations of periodic polarity-inverted GaN waveguides <u>R. Katayama</u> , Y. Fukuhara, M. Kakuda, S. Kuboya, K. Onabe, and T. Matsuoka <i>Inst. for Mater. Res., Tohoku Univ, Japan; PRESTO, Japan Sci. and Technol. Agency, Japan; Dept. of Adv. Mater. Sci., Univ. of Tokyo, Japan</i>	103
Th-5o	10:30 - 10:45 Carrier dynamics in semipolar GaN / Al_{0.5}Ga_{0.5}N (11-22) nanostructures <u>A. Kahouli</u> , J. Brault, B. Damilano, N. Kriouche, M. Leroux, P. Vennéguès, P. De Mierry, P. Valvin, C. Brimont, T. Guillet, and J. Massies <i>Centre de Recherche sur l'hétéro-Epitaxie et ses Applications, Centre National de la Recherche Scientifique, France; Université de Nice Sophia Antipolis, France; Laboratoire Charles Coulomb UMR 5221, Université Montpellier 2- CNRS, France</i>	104
<u>Coffee Break</u>		10:45 - 11:15
Session 12 “III-N Growth on Si” Chair: R. Dupuis		11:15 - 13:00
Th-2i	11:15 - 11:45 Growth of quantum wire III-N nanostructures <u>B. Daudin</u> , C. Bougerol, B. Gayral, K. Hestroffer, M. Kociak, C. Leclere, Z. Mahfoud, H. Renevier, D. Sam Giao, G. Tourbot <i>Institut Néel/CNRS-Univ. J. Fourier and CEA Grenoble/INAC/SP2M,; Laboratoire de Physique des Solides, CNRS UMR8502, Université Paris-Sud; Laboratoire des Matériaux et du Génie Physique, Grenoble INP - MINATEC; CEA, LETI, MINATEC, France</i>	105
Th-6o	11:45 - 12:00 Single-crystal GaN on SiO₂ for monolithic integration with CMOS J. Song, B. Leung, Y. Zhang, and <u>J. Han</u> <i>Department of Electrical Engineering, Yale University, USA</i>	106
Th-7o	12:00 - 12:15 MOVPE growth of In_xGa_{1-x}N (x~0.5) on Si(111) substrates with a pn junction on the surface <u>A. Yamamoto</u> , D. Hironaga, A. Mihara, Y. Muramatsu, K. Sugita, A.G. Bhuiyan, A. Hashimoto, N. Shigekawa, and N. Watanabe <i>University of Fukui, Japan; Osaka City University, Japan; NTT Photonics Laboratories, Japan</i>	107
Th-8o	12:15 - 12:30 AlGaIn/GaN Heterostructures on Silicon (111) Substrates via Rare Earth Oxide Buffer Layers F. Erdem Arkun, R. Dargis, R. Roucka, A. Clark, <u>M. L. Lebbby</u> <i>Translucent Inc., USA</i>	108

		page
Th-3i	12:30 - 13:00 Growth of GaN-based heterostructures on silicon substrates <u>F. Semond</u> , M. J. Rashid, G. Gommé, S. Sergent, Y. Cordier, B. Damilano, E. Frayssinet, J.-Y. Duboz, M. Leroux, and J. Massies <i>CRHEA-CNRS, France</i>	109
	<u>Lunch</u>	13:00 - 14:15
	Session 13 “Defects and impurities” Chair: T. Shubina	14:15 - 15:45
Th-4i	14:15 - 14:45 Luminescence dynamics in undoped and Mg-doped III-Nitrides <u>B. Monemar</u> , G Pozina, P. P. Paskov, J. P. Bergman, S Khromov <i>Department of Physics, Chemistry and Biology, Linköping University, Sweden</i>	110
Th-9o	14:45 - 15:00 Direct measurement of the Mg lattice site location in group III-Nitrides <u>L. M. Amorim</u> , U. Wahl, S. Decoster, L. M. C. Pereira, J. G. Correia, D. J. Silva, K. Temst and A. Vantomme <i>Instituut voor Kern en Stralingsfysica, -KU Leuven, Belgium; Instituto Tecnológico e Nuclear, Portugal; ISOLDE-CERN, Switzerland; Universidade do Porto, Portugal</i>	111
Th-10o	15:00 - 15:15 Microstructure of Mg doped GaNAs Alloys <u>Z. Liliental-Weber</u> , R. dos Reis, S.V. Novikov, K.M. Yu, A.X. Levander, O.D. Dubon, J. Wu, W. Walukiewicz, and C.T. Foxon <i>Materials Science Division, Lawrence Berkeley National Laboratory, USA; School of Physics and Astronomy, Univ. of Nottingham, UK; Department of Materials Science & Engineering, University of California, USA</i>	112
Th-11o	15:15 - 15:30 Nitrogen Rich Defects in InN <u>K.S.A. Butcher</u> , D. Alexandrov, V. Georgiev, D. Georgieva, P. P.-T.Chen and J. E. Downes <i>Semiconductor Research Laboratory, Department of Electrical Engineering, Lakehead University Canada; Meaglow Ltd, Canada; Department of Physics and Astronomy, Faculty of Science, Macquarie University, Australia</i>	113
Th-12o	15:30 - 15:45 Suppression of twin formation for the growth of InN(10$\bar{1}$3) on GaAs(110) by metalorganic vapor phase epitaxy <u>H. Murakami</u> , S. Takenaka, T. Hotta, Y. Kumagai and A. Koukitu <i>Department of Applied Chemistry, Tokyo University of Agriculture and Technology, Japan</i>	114
	<u>Coffee Break</u>	15:45 - 16:15

		page
Session 14 “3D Nanostructures and Devices”	16:15 - 18:00	
	Chair: B. Daudin	
Th-5i	16:15 - 16:45 3D GaN Core-Shell Structures for Solid State Lighting <u>A. Waag</u> <i>Institute of Semiconductor Technology, Braunschweig University of Technology, Germany</i>	115
Th-13o	16:45 - 17:00 Efficiency Improvement of Nonpolar a-plane GaN Light Emitting Diodes by Controlled Integration of Silica Nano-spheres S.H. Park, J. Jang, K. Joo, D. Moon, D.-J. You, J. Park, J. Xu, Y. Nanishi, and <u>E. Yoon</u> <i>Department of Materials Science and Engineering, Seoul National University, Korea; WCU Hybrid Materials Program, Department of Materials Science and Engineering, Seoul National University, Korea; Department of Nano Science and Technology, Graduate School of Convergence Science and Technology, Seoul National University, Korea; Department of Electronic Engineering, Hanyang University, Korea; Department of Engineering, Brown University, USA; Department of Photonics, Ritsumeikan University, Japan</i>	116
Th-14o	17:00 - 17:15 Improved performance of blue light emitting diodes with embedded multiple layers of dielectric spheres Y.J. Park, J.H. Kang, H.Y. Kim, H.K. Kim, N.Han, M.Han, B.D.Ryu, K.B. Ko, and <u>C.-H. Hong</u> <i>Semiconductor Physics Research Center, School of Semiconductor and Chemical Engineering, Chonbuk National University, South Korea</i>	117
Th-15o	17:15 - 17:30 Kinetics of Initial Stages of Growth of thin films of nano-structured GaN on Al₂O₃ (0001) <u>S. M. Shivaprasad</u> <i>Jawaharlal Nehru Centre for Advanced Scientific Research, India</i>	118
Th-6i	17:30 - 18:00 Ultra High Power and RF operation of GaN/InGaN Heterojunction Bipolar Transistors on Free Standing GaN Substrates Z. Lochner, H.J. Kim, Y.-C. Lee, Y. Zhang, S. Choi, J.-H. Ryou, S.-C. Shen, P.D. Yoder, and <u>R.D. Dupuis</u> <i>Center for Compound Semiconductors and School of Electrical and Computer Engineering, Georgia Institute of Technology, USA</i>	119
	<u>Closing remarks</u>	18:00 - 18:20

POSTER SESSION I

Monday, July 16

	18:00- 19:30	page
Mo-1p	AlGaN layer structures for deep UV emitters on laterally overgrown AlN <u>A. Knauer</u> , V. Kueller, U. Zeimer, C. Reich, M. Weyers and M. Kneissl <i>Ferdinand-Braun-Institut, Germany; Technische Universität Berlin, Institute of Solid State Physics, Germany</i>	120
Mo-2p	CHVPE growth of AlGaN-based UV LED <u>S. Kurin</u> , A. Antipov, I. Barash, A. Roenkov, H. Helava, Yu. Makarov <i>Nitride Crystals Ltd., Russia; Nitride Crystals Inc., USA</i>	121
Mo-3p	Development of Micro-Plasma Excited Deep Ultraviolet Light Emitting Device (MIPE-DUV Light Emitter) using AlGaN grown by Metal Organic Chemical Vapor Phase Epitaxy <u>N. Kurose</u> , M. Kurouchi, M. Takeuchi, Y. Aoyagi <i>Ritsumeikan University, Japan</i>	122
Mo-4p	Fabrication of 2'' freestanding GaN substrates by HVPE using low temperature buffer layer on sapphire <u>C. Hemmingsson</u> , G. Pozina, and B. Monemar <i>Department of Physics, Chemistry and Biology, Linköping University, Sweden</i>	123
Mo-5p	The effect of surface state of the seed substrate on the growth of GaN single crystal by Na flux method <u>M. Honjo</u> , T. Fujimori, K. Masumoto, H. Imabayashi, A. Kitamoto, H. Takazawa, Y. Todoroki, D. Matsuo, K. Murakami, M. Maruyama, M. Imade, M. Yoshimura, T. Sasaki, Y. Mori <i>Osaka University, Japan</i>	124
Mo-6p	Coalescence of GaN nanorods for high quality templates M. Hugues, P.A. Shields, E. Beraudo, <u>F. Semond</u> , M. Cooke, M. Dineen, M. Mandl, P. Rode, M. Strassburg, F. Causa, D.W.E. Allsopp, and J. Zúñiga-Pérez <i>CRHEA-CNRS, France; Dept. of Electronic and Electrical Engineering, University of Bath, UK; Oxford Instruments Plasma Technology, UK; OSRAM Opto Semiconductors GmbH, Germany</i>	125
Mo-7p	Stress distribution in 12µm thick continuous GaN grown on patterned Si substrate T. Hossain, E. Frayssinet, S. Chenot, M. Leroux, B. Damilano, F. Demangeot, A. Ponchet, M. J. Rashid, <u>F. Semond</u> and Y. Cordier <i>CRHEA-CNRS, France ; Physics Department, University of Nice-Sophia Antipolis, France ; CEMES-CNRS UPR-8011, France</i>	126

		page
Mo-8p	The coalescence growth of GaN crystals on point seed crystals using Na flux method <u>M. Imanishi</u> , K. Masumoto, K. Murakami, H. Imabayashi, H. Takazawa, Y. Todoroki, D. Matsuo, M. Maruyama, M. Imade, M. Yoshimura, and Y. Mori <i>Department of Electrical, Electronic and Information Engineering, Osaka University, Japan</i>	127
Mo-9p	Solid-source solution growth (3SG) method for bulk AlN growth <u>Y. Kangawa</u> , N. Kuwano, B. M. Epelbaum, and K. Kakimoto <i>RIAM, Kyushu Univ., Japan; PRESTO, JST, Japan; KASTEC, Kyushu Univ., Japan; Department of Materials Science 6, University of Erlangen, Germany</i>	128
Mo-10p	Growth of Bulk AlN using Alumina Reduction Method <u>M. Kato</u> , M. Ohtsuka and H. Fukuyama <i>Institute of Multidisciplinary Research for Advanced Materials (IMRAM), Tohoku University, Japan</i>	129
Mo-11p	Molybdenum as local growth inhibitor in ammonia based epitaxy processes <u>M. Klein</u> , and F. Scholz <i>Institute of Optoelectronics, Ulm University, Germany</i>	130
Mo-12p	Gallium Nitride Crystal Growth Insitu Monitoring Techniques Using Bulk Material Optical Properties <u>Y. Lacroix</u> <i>YSystems Ltd., Japan</i>	131
Mo-13p	Ammonothermal crystal growth of GaN using an NH₄Br mineralizer <u>K. Qiao</u> , Q. Bao, S. F. Chichibu, T. Ishiguro, C. Yokoyama <i>CRHEA-CNRS, Valbonne, France; LPN-CNRS, Marcoussis, France; Institute of Multidisciplinary Research for Advanced Materials, Tohoku University, Japan</i>	132
Mo-14p	N-type doping of HVPE-grown GaN boules using dichlorosilane <u>M. Weyers</u> , E. Richter, U. Zeimer, C. Netzell, and G. Tränkle <i>Ferdinand-Braun-Institut Leibniz-Institut fuer Hoechstfrequenztechnik, Germany</i>	133
Mo-15p	Free-Standing GaN Wafer by HVPE with a Pit-Induced Buffer layer <u>T. Sato</u> , T. Goto, T. Yao, A. Sato and H. Gotoh <i>Center for Interdisciplinary Research, Tohoku University, Japan; Tsukuba R&D Center, AETech Co., LTD, Japan</i>	134
Mo-16p	GaN growth modes and corresponding macrodefects <u>V.V. Voronenkov</u> , N.I. Bochkareva, R.I. Gorbunov, P.E. Latyshev, Y.S. Lelikov, Y.T.Rebane, A.I. Tsyuk, A.S. Zubrilov and Y.G. Shreter <i>St.-Petersburg State Polytechnical University, Russia; Ioffe Physical Technical Institute, Russia; Fock Institute of Physics St. Petersburg State University, Russia</i>	135

		page
Mo-17p	Powder synthesis of GaN using Ga metal as a starting material <u>C. Yokoyama</u> , Q. Bao, S. F. Chichibu, T. Ishiguro, K. Qiao <i>Institute of Multidisciplinary Research for Advanced Materials, Tohoku University, Japan</i>	136
Mo-18p	Impact of SiN_x passivation on the maximum current of AlGa_N/Ga_N HEMTs <u>M. Azize</u> , D. Piedra and T. Palacios <i>Microsystems Technology Laboratories, Massachusetts Institute of Technology, USA</i>	137
Mo-19p	Growth temperature effect on two-dimensional electron gas density in AlGa_N/Ga_N heterostructure <u>J.-T. Chen</u> , I. Booker, I.G. Ivanov, A. Lundskog, U. Forsberg, and E. Janzén <i>Department of Physics, Chemistry, and Biology, Linköping University, Sweden</i>	138
Mo-20p	Experimental and theoretical study of electrical, optical, and thermal processes in high-power InGa_N/Ga_N LEDs <u>A.E. Chernyakov</u> , A.L. Zakgeim, K.A. Bulashevich, and S.Yu. Karpov <i>Submicron Heterostructures for Microelectronics Research & Engineering Center, RAS, Russia, STR Group – Soft-Impact, Ltd., Russia</i>	139
Mo-21p	Peculiarities of III-Nitride heterostructure design and MBE growth conditions for two-dimensional electron gas and acoustic waves based devices D. Krasovitsky, <u>A. Dudin</u> , S. Kokin, N. Katsavets, and V. Chaly <i>Svetlana-Rost, JSC, Russia</i>	140
Mo-22p	Manipulation of Carrier Distributions in III-Nitride Visible Light-Emitting Diodes by Hole Injection Layer and Electron Blocking Layer J. Kim, M.-H. Ji, R. Gong, S. Choi, N. Sebkhi, Md.M. Satter, P.D. Yoder, J.-H. Ryou, <u>R.D. Dupuis</u> , A. Fischer, and F.A. Ponce <i>Center for Compound Semiconductors and School of Electrical and Computer Engineering, Georgia Institute of Technology, USA; Department of Physics, Arizona State University, USA</i>	141
Mo-23p	Comparative study of In_xGa_{1-x}N/GaN and In_xGa_{1-x}N/Mg_yZn_{1-y}O High Efficiency Solar Cells <u>A. Kadri</u> , K. Zitouni <i>LEMOP Laboratory, Physics Department, University of Oran (Es-Senia), Algeria</i>	142
Mo-24p	The description of quantum states in nanostructures Ga_N / InGa_N using the envelope functions. The role of boundary conditions <u>G. Karavaev</u> , V. Chernyshov, A.Razzhuvalov <i>Tomsk State University, Russia; Tomsk Polytechnic University, Russia</i>	143

		page
Mo-25p	Selectively-sensitive MSM-photodetectors based on AlGaIn/AlN and ZnCdS/GaP heterostructures S. Averin, <u>P. Kuznetsov</u> , V. Zhitov, L. Zakharov, N. Alkeev, N. Gladisheva <i>Kotel'nikov FIRE RAS, Russia; Federal State Unitary Enterprise "SPE"Pulsar", Russia</i>	144
Mo-26p	C-doped asymmetric cubic AlN/GaN multi quantum well structures for high electrical isolation to 3C-SiC substrates <u>A. Zado</u> and D.J. As <i>University of Paderborn, Faculty of Science, Department of Physics, Germany</i>	145
Mo-27p	Nondestructive method for evaluation of electrical parameters of AlGaIn/GaN HEMT heterostructures B. Paszkiewicz, M. Wosko, <u>R. Paszkiewicz</u> , M. Tlaczala <i>Wroclaw University of Technology, Faculty of Microsystem Electronics and Photonics, Poland</i>	146
Mo-28p	Determination of recombination lifetimes in InGaIn/GaN based LEDs by differential lifetime analysis <u>L. Riuttanen</u> , S. Suihkonen, P. Kivisaari, J. Oksanen and M. Sopanen <i>Department of Micro- and Nanosciences, Aalto University, Finland; Department of Biomedical Engineering and Computational Science, Aalto University, Finland</i>	147
Mo-29p	High signal-to-noise ratio visible-blind photodetector by using slightly Mg-doping InGaIn <u>L. Sang</u> , M. Liao, Y. Koide, and M. Sumiya <i>National Institute for Materials Science, Japan</i>	148
Mo-30p	Extended defects system as a main source of non-radiative recombination in InGaIn/GaN LEDs N. Averkiev, A. Chernyakov, M. Levinshtein, P. Petrov, N. Shmidt, <u>E. Shabunina</u> <i>Ioffe Physical Technical Institute, Russia</i>	149
Mo-31p	Mechanisms behind efficiency droop and degradation in InGaIn/GaN LEDs A. Greshnov, A. Chernyakov, M. Levinshtein, A.L. Zakgeim, <u>N. Shmidt</u> , E. Shabunina <i>Ioffe Physical Technical Institute, Russia</i>	150
Mo-32p	Capacitance-Voltage and Admittance Investigations of InGaIn/GaN MQW LEDs: Frequency Dependence <u>O.A. Soltanovich</u> and E.B. Yakimov <i>Institute of Microelectronics Technology RAS, Russia</i>	151
Mo-33p	Ab initio modeling of AlN/GaN multi quantum wells <u>Pawel Strak</u> , Pawel Kempisty, Stanislaw Krukowski <i>Institute of High Pressure Physics, Polish Academy of Sciences, Poland; Interdisciplinary Centre for Modelling, University of Warsaw, Poland</i>	152

		page
Mo-34p	p-type AlInN for Lattice-Matched Nitride Devices <u>Y. Taniyasu</u> , J.-F. Carlin, A. Castiglia, R. Butté, and N. Grandjean <i>NTT Basic Research Laboratories, NTT Corporation, Japan; Ecole Polytechnique Fédérale de Lausanne (EPFL), Switzerland</i>	153
Mo-35p	Optical and electrical characteristics of AlGaN-based UV LEDs <u>S. Tarasov</u> , E. Menkovich, I. Lamkin, S. Kurin, A. Antipov, I. Barash, A. Roenkov, H. Helava, Yu. Makarov <i>Saint-Petersburg Electrotechnical University "LETI", Russia; Nitride Crystals Ltd., Russia; Nitride Crystals Inc., USA</i>	154
Mo-36p	Advantage of tapered and graded AlGaN electron blocking layer in InGaN-based blue laser diodes <u>W. Yang</u> , D. Li, N. Liu, L. Liu, Z. Chen and X. Hu <i>State Key Laboratory for Artificial Microstructure and Mesoscopic Physics, School of Physics, Peking University, People's Republic of China</i>	155
Mo-37p	Band modification in InGaN/GaN multiple quantum wells sandwiched by symmetrical thin low indium-InGaN layers <u>J.Z. Li</u> , Y.B. Tao, Z.Z. Chen, X.Z. Jiang, X.X. Fu, S.Y. Wang, T.J. Yu, G.Y. Zhang <i>State Key Laboratory for Artificial Microstructures and Mesoscopic Physics, School of Physics, Peking University, People's Republic of China</i>	156
Mo-38p	Fabrication and characterization of 80µm-thick free-standing homoepitaxial GaN-based blue LED <u>J.Z. Li</u> , Y.B. Tao, Z.Z. Chen, X.Z. Jiang, X.X. Fu, S. Jiang, Z. Hu, T.J. Yu, G.Y. Zhang <i>State Key Laboratory for Artificial Microstructures and Mesoscopic Physics, School of Physics, Peking University, People's Republic of China</i>	157
Mo-39p	Growth kinetics of III-nitrides by MOVPE and MBE <u>N.A.K. Kaufmann</u> , D. Martin, J.-F. Carlin and N. Grandjean <i>ICMP-LASPE, Ecole Polytechnique Fédéral de Lausanne, Switzerland</i>	158
Mo-40p	Gas phase mass transport and surface diffusion in extreme lateral overgrowth for cantilever sensor applications <u>Q. Jiang</u> , M. Edwards, P. Shields, C. R. Bowen, C.W.E. Allsopp, W. N. Wang, L. Tóth, and B. Pécz <i>Department of Electrical and Electronic Engineering, University of Bath, UK; Ins. for Tech. Phys. Mat. Sci., Hungary</i>	159
Mo-41p	Investigation of self-terminated growth of in-situ deposited SiN_x nano-mask <u>T. Markurt</u> , L. Lymperakis, J. Neugebauer, T. Schulz, T. Remmele, P. Drechsel, P. Stauss, E. Rotunno, V. Grillo and M. Albrecht <i>Leibniz Institute for Crystal Growth, Germany; Max-Planck Institut für Eisenforschung, Germany; OSRAM Opto Semiconductors, Germany; IMEM-CNR, Italy</i>	160

		page
Mo-42p	Influence of reactor pressure on InGaN/GaN MQW growth and properties <u>A.V. Sakharov</u> , A.E. Nikolaev, W.V. Lundin, S.O. Usov, E.E. Zavarin, and A.F. Tsatsulnikov <i>Ioffe Physical Technical Institute, Russia</i>	161
Mo-43p	Influence of growth process scheme and parameters dispersion on the properties of AlGaIn/GaN heterostructures <u>M. Wośko</u> , B. Paszkiewicz, R. Paszkiewicz, and M. Tłaczała <i>Faculty of Microsystem Electronics and Photonics, Wrocław University of Technology, Poland</i>	162
Mo-44p	Observation of Exciton-Phonon Interaction in AlGaIn/AlN Quantum Wells <u>R.G. Banal</u> , M. Funato, and Y. Kawakami <i>Department of Electronic Science and Engineering, Kyoto University, Japan</i>	163
Mo-45p	Optical anisotropy investigation from strained (In_xGa_{1-x}N/GaN) layers grown on (polar) c-sapphire <u>Y. Basma</u> , S.M.C. Miranda, N. Franco, E. Alves, K. Lorenz, Iman Roqan <i>Kaust, Saudi Arabia; Instituto Tecnológico e Nuclear, Portugal</i>	164
Mo-46p	Microphotoluminescence studies of phase separation in InGaIn solid alloys <u>K.G. Belyaev</u> , M.V. Rakhlin, V.N. Jmerik, A.M. Mizerov, Ya.V. Kuznetsova, M.V. Zamoryankaya, S.V. Ivanov, and A.A. Toropov <i>Ioffe Physical Technical Institute, Russia</i>	165
Mo-47p	Cathodoluminescence study of InGaIn quantum well grown on GaN annular structure by metal-organic chemical vapor deposition Y.-H. Ko and <u>Y.-H. Cho</u> , B. Leung, J. Song, C. Yerino, and J. Han <i>Department of Physics and Graduate School of Nanoscience & Technology (WCU), Korea Advanced Institute of Science and Technology (KAIST), Republic of Korea; Department of Electrical Engineering, Yale University, USA</i>	166
Mo-48p	Growth Mechanisms of Catalyst-free and Mask-free Heteroepitaxial GaIn Nanorods and Microrods on Si (111) and Si (100) Substrates Grown by MOVPE S.-M. Ko, J.-H. Kim, Y.-H. Ko, Y. H. Chang, Y.-H. Kim, J. M. Yoon, J. Y. Lee, and <u>Y.-H. Cho</u> <i>Department of Physics and Graduate School of Nanoscience & Technology (WCU), Korea Advanced Institute of Science and Technology (KAIST), Republic of Korea; Graduate School of Nanoscience and Technology (WCU), KAIST, Republic of Korea; Department of Materials Science and Engineering, KAIST, Republic of Korea</i>	167

		page
Mo-49p	Lattice dynamics of short period GaN/AlN superlattices. Theory and experiment <u>V.Yu. Davydov</u> , E.M. Roginskii, A.N. Smirnov, Yu.E. Kitaev, M.A. Yagovkina, R.N. Kyutt, M.M. Rojavskaja, E.E. Zavarin and W.V. Lundin, M.B. Smirnov <i>Ioffe Physical Technical Institute, Russia; Fock Institute of Physics, St. Petersburg State University, Russia</i>	168
Mo-50p	Polarization effects in light emission from strained AlGaIn-based heterostructures M. Durnev and <u>S. Yu. Karpov</u> <i>STR Group – Soft Impact, Ltd., Russia</i>	169
Mo-51p	Time-resolved photoluminescence studies on HVPE freestanding GaN substrates exhibiting record-long positron diffusion length <u>K. Hazu</u> , Y. Ishikawa, M. Tashiro, H. Namita, S. Nagao, K. Fujito, A. Uedono, and S. F. Chichibu <i>Institute of Multidisciplinary Research for Advanced Materials, Tohoku University, Japan; Mitsubishi Chemical Corporation, Optoelectronics Department, Japan; Institute of Applied Physics, University of Tsukuba, Japan</i>	170
Mo-52p	Full molar fractional Raman spectra from RF-MBE grown In_xAl_{1-x}N alloys (0 < x < 1) <u>K. Kodama</u> , H. Harima, A. Yamamoto, and A. Hashimoto <i>Department of Electrical & Electronics Engineering, Graduate School of Engineering, University of Fukui, Japan; Department of Electronics and Information Science, Kyoto Institute of Technology, Japan</i>	171
Mo-53p	Unstable luminescence of nitrides under electron-beam irradiation <u>Ya. Kuznetsova</u> , and M. Zamoryanskaya <i>Ioffe Physical Technical Institute, Russia</i>	172
Mo-54p	Temperature-resolved photoluminescence of nonpolar InGaIn/GaN multiple quantum well heterostructures grown on LiAlO₂ <u>E.V. Lutsenko</u> , M. V. Rzhetski, V.N. Pavlovskii, G.P. Yablonskii, C. Mauder, B. Reuters, H. Kalisch, M. Heuken, A. Vescan <i>Stepanov Institute of Physics, National Academy of Sciences of Belarus, Belarus; GaN Device Technology, RWTH Aachen University, Germany; AIXTRON SE, Germany</i>	173
Mo-55p	The effect of the magnetic field on the micro-photoluminescence spectra of the InGaIn/GaN MQW structures doped with Eu, Sm and Eu+Sm <u>M.M. Mezdrogina</u> , E.S. Moskalenko and Yu.V.Kozhanova <i>Ioffe Physical-Technical Institute, Russia; Polytechnical University, Russia</i>	174

		page
Mo-56p	Cubic III-nitride coupled quantum wells towards unipolar optically pumped lasers <u>C. Mietze</u> , M. Bürger, S. Sakr, M. Tchernycheva, F.H. Julien, and D.J. As <i>University of Paderborn, Department of Physics, Germany; Institut d'Electronique Fondamentale, UMR 8622 CNRS, University Paris-Sud 11, France</i>	175
Mo-57p	Confocal micro-Raman spectroscopy in-depth and across the cleaved edge of vertically designed GaN Gunn-diode structure A.E. Belyaev, <u>V.V. Strelchuk</u> , A.S. Nikolenko, V.V. Gomonovych, Yu.I. Mazur, M.E. Ware, E.A. DeCuir Jr., G.J. Salamo <i>V. Lashkaryov Institute of Semiconductor Physics National Academy of Sciences of Ukraine, Ukraine; Department of Physics, University of Arkansas, USA</i>	176
Mo-58p	Confocal Raman and photoluminescence microscopy of light-emitting heterostructures with multiple InGaN/GaN quantum wells A.E. Belyaev, <u>V.V. Strelchuk</u> , A.F. Kolomys, E.A. Avramenko, A.S. Romanyuk, R.V. Konakova, B.S. Yavich <i>V. Lashkaryov Institute of Semiconductor Physics National Academy of Sciences of Ukraine, Ukraine; Svetlana Optoelectronics JSC, Russia</i>	177
Mo-59p	Peculiarities of photoluminescence efficiency dependence on excitation intensity in GaN/Al₂O₃ epilayers E.V. Lutsenko, M.V. Rzhetski, V.N. Pavlovskii, G.P. Yablonskii, <u>I. Reklaitis</u> , A. Kadys, T. Malinauskas, S. Nargelas, K. Jarašiūnas, and A. Žukauskas <i>B.I. Stepanov Institute of Physics of NAS of Belarus, Belarus; Institute of Applied Research, Vilnius University, Lithuania</i>	178
Mo-60p	Dramatic thermal quenching of photoluminescence in Zn-doped GaN <u>M.A. Reshchikov</u> <i>Physics Department, Virginia Commonwealth University, USA</i>	179
Mo-61p	Electrical and optical properties of bulk GaN substrates studied by Kelvin probe and photoluminescence M.A. Foussekis, J.D. McNamara, A.A. Baski, and <u>M.A. Reshchikov</u> , X. Li, V. Avrutin, and H. Morkoç, J.H. Leach, T. Paskova, K. Udvary, and E. Preble <i>Physics Department, Virginia Commonwealth University, USA; Department of Electrical and Computing Engineering, Virginia Commonwealth University, USA; Kyma Technologies, Inc., USA</i>	180

		page
Mo-62p	Properties of MOCVD AlGaIn/GaN heterostructures grown on polar and non-polar bulk GaN substrates <u>M. Rudziński</u> , R. Kudrawiec, R. Kucharski, R. Dwiliński, W. Strupiński <i>Institute of Electronic Materials Technology, Poland; Institute of Physics, Wrocław University of Technology, Poland; 3AMMONO sp. z.o.o., Poland</i>	181
Mo-63p	Quantum-confined Stark Effect and Carriers Localization in Al_{0.3}Ga_{0.7}N/Al_{0.4}Ga_{0.6}N Quantum Wells of Different Morphology <u>E. Shevchenko</u> , V. Jmerik, A. Mizerov, D. Nechaev, A. Sitnikova, S. Ivanov, and A. Toropov <i>Ioffe Physical-Technical Institute, Russia</i>	182
Mo-64p	Optical saturation of intraband absorption of GaN/AlN quantum-dot waveguides at 1.55 μm <u>S. Valdueza-Felip</u> , L. Monteagudo-Lerma, F. B. Naranjo, P. Corredera, L. Rapenne, E. Sarigiannidou, G. Strasser, E. Monroy and M. González-Herráez <i>GRIFO, Dpto. Electrónica, Universidad de Alcalá, Spain; Instituto de Física Aplicada, CSIC, Spain; INP-Grenoble/Minatoc, France; Zentrum für Mikro- und Nanostrukt., TU Vienna, Austria; CEA-Grenoble, INAC/SP2M/NPSC, France</i>	183
Mo-65p	Continuous wave OSL in bulk AlN single crystals <u>A.S. Vokhmintsev</u> , I.A. Weinstein, D.M. Spiridonov, E.Yu. Belousova <i>Ural Federal University, REC «Nanomaterials and Nanotechnologies», Russia</i>	184
Mo-66p	Effect of the nitridation on sapphire surface polaritons <u>K.S. Zhuravlev</u> , T.V. Malin, N.N. Novikova and V.A. Yakovlev <i>A.V.Rzhanov Institute of Semiconductor Physics, Siberian Branch of the Russian Academy of Sciences, Russia; Institute for Spectroscopy of the Russian Academy of Sciences, Russia</i>	185
Mo-67p	Characterization of MBE grown AlGaIn layers heavily doped using silane <u>K.S. Zhuravlev</u> , D.Yu. Protasov, T.V. Malin, V.Yu. Davydov, A.N. Smirnov and R.N. Kyutt, A.V. Spirina, V.I. Solomonov <i>A.V.Rzhanov Institute of Semiconductor Physics, Siberian Branch of the Russian Academy of Sciences, Russia; Ioffe Physical Technical Institute, of the Russian Academy of Sciences, Russia; Institute of Electrophysics, Ural Division of the Russian Academy of Sciences, Russia</i>	186
Mo-68p	Characterization of Nitrides based LED materials using dynamic SIMS <u>A. Merkulov</u> <i>CAMECA, France</i>	187

		page
Mo-69p	Self-organized defect control during GaN homo-epitaxial growth on nano-structured substrates <u>M. Mynbaeva</u> , A. Sitnikova, A. Nikolaev, K. Vinogradova, K. Mynbaev, V. Nikolaev <i>Ioffe Physical Technical Institute, Russia; Perfect Crystals LLC, Russia</i>	188
Mo-70p	Low Energy Electron Beam Induced Ga-Vacancy Activation in Gallium Nitride Films <u>H. Nykänen</u> , S. Suihkonen, M. Sopanen and F. Tuomisto <i>Department of Micro- and Nanosciences, Aalto University, Finland;</i> <i>Department of Applied Physics, Aalto University, Finland</i>	189
Mo-71p	Doping effect on optical properties of freestanding bulk GaN <u>G. Pozina</u> , S. Khromov, C. Hemmingsson, and B. Monemar <i>Department of Physics, Chemistry and Biology, Linköping University, Sweden</i>	190
Mo-72p	Role of extended defects in the transformation of InGaN/GaN multiple quantum well structure optical properties under low energy electron beam irradiation P.S. Vergeles, N.M. Schmidt, <u>E.B. Yakimov</u> <i>Institute of Microelectronics Technology RAS, Russia; Ioffe Physical Technical Institute, Russia</i>	191
Mo-73p	HVPE growth of GaN in the semipolar direction on planar Si(210) <u>V.N. Bessolov</u> , E.V. Konenkova, M.P. Shcheglov, Sh. Sharofidinov, S.A. Kukushkin, A.V. Osipov, V.I. Nikolaev <i>A.F.Ioffe Physico-Technical institute, Russia; Institute of Problems of Mechanical Engineering, Russia; «Perfect crystals» LC, Russia</i>	192
Mo-74p	CHVPE GaN growth on SiC and Si <u>H. Helava</u> , A. Antipov, I. Barash, A. Roenkov, S. Kurin, S. Nagalyuk, D. Litvin, A. Vasiliev, Yu. Makarov <i>Nitride Crystals Inc., USA; Nitride Crystals Ltd., Russia</i>	193
Mo-75p	Growth of GaN Nanorods on Si (111) substrate by Reactive Magnetron Sputter Epitaxy using a Liquid Ga as a Sputtering Target <u>M. Junaid</u> , J. Palisaitis, Y.-T. Chen, C.-L. Hsiao, P. O. Å. Persson, L. Hultman, and J. Birch <i>Department of Physics, Chemistry and Biology (IFM), Linköping University, Sweden</i>	194
Mo-76p	Improvement in crystalline quality of GaN prepared by pulsed sputtering by the use of SiN_x islands <u>T. Kondo</u> , J. Ohta, S. Inoue, A. Kobayashi, and H. Fujioka <i>Institute of Industrial Science, Univ. of Tokyo, Japan; Core Research for Evolutional Science and Technology (CREST), Japan Science and Technology Agency (JST), Japan</i>	195

		page
Mo-77p	Effect of silicon-nitride intermediate layer on self-aligned GaN nanorod growth on Si(111) surface <u>Praveen Kumar</u> and S. M. Shivaprasad <i>Jawaharlal Nehru Centre for Advanced Scientific Research, India;</i> <i>Institute for Systems based on Optoelectronics and Microtechnology,</i> <i>Technical University of Madrid, Spain</i>	196
Mo-78p	Growth of the thick GaN layers on Si substrates <u>V.A. Kureshov</u> , A.V. Mazalov, D.R. Sabitov, A.A. Padalitsa, A.A. Marmalyuk, R.Kh. Akchurin <i>Sigm Plus Co., Russia; Moscow University of Fine Chemical Technology,</i> <i>Russia</i>	197
Mo-79p	Activity modulation MEE growth of 2H-AlN on Si(111) using double buffer layer grown by PA-MBE <u>T. Ohachi</u> , Y. Yamamoto, O. Ariyada, Y. Sato, S. Yoshikado and M. Wada <i>Department of Electrical Engineering, Doshisha University Japan; ARIOS</i> <i>Inc., Japan</i>	198
Mo-80p	MOVPE growth of GaN wires on Si(111) for LED applications <u>D. Salomon</u> , A. Dussaigne, D. Sam-Giao, B. Amstatt, C. Bougerol, A.L. Bavencove, P. Gilet, P. Ferret, C. Durand, and J. Eymery <i>Equipe mixte CEA-CNRS-UJF "Nanophysique et semiconducteurs",</i> <i>SP2M, UMR-E CEA / UJF-Grenoble 1, INAC, France; CEA-Leti,</i> <i>MINATEC campus, France; Institut Néel-CNRS, France</i>	199
Mo-81p	Self-induced MBE growth of GaN nanowires on Si substrates with thin Al₂O₃ buffer layer <u>Z.R. Zytkeiwicz</u> , A. Wierzbicka, M. Sobanska, K. Klosek, A. Reszka, A. Cabaj, G. Tchutchulashvili, and S. Gieraltowska <i>Institute of Physics, Polish Academy of Sciences, Poland</i>	200

POSTER SESSION II

Tuesday, July 17

	18:00- 19:30	page
Tu-1p	InGaN-based heterostructures for solar cells <u>A. Mukhtarova</u> , C. Durand, H. Mariette, and J. Eymery <i>CEA-CNRS group « Nanophysique et Semiconducteurs », CEA/INAC/SP2M-Grenoble, France</i>	201
Tu-2p	Growth and deep level defect evaluation of InGaN films for the application of photovoltaic devices <u>M. Sumiya</u> , L. W. Sang, M. Lozac'h and Y. Nakano <i>Wide-gap Material Group, National Institute for Materials Science, Japan; JST-Advance Low Carbon Technology Research and Development Program, Japan; International Center for Young Scientists, National Institute for Materials Science, Japan; Institute of Science and Technology Research, Chubu University, Japan</i>	202
Tu-3p	Fabrication of Zn-doped GaN-Pillars as Potential Sources for Single-Photon Emission A. Behrends, M. Mohajerani, <u>A. Bakin</u> and A. Waag, S. Peters, H. Hofer, W. Schmunk and S. Kück, S. Shvarkov and A.D. Wieck, M.A. Reshchikov <i>Institute of Semiconductor Technology, Technische Universität Braunschweig, Germany; Physikalisch Technische Bundesanstalt (PTB), Braunschweig, Germany; Lehrstuhl für Angewandte Festkörperphysik, Ruhr-Universität Bochum, Germany; Physics Department, Virginia Commonwealth University, USA</i>	203
Tu-4p	Structural characterization of epitaxial GaN wires: polarity determination and dislocation bending <u>P. M. Coulon</u> , O. Tottereau, B. Alloing, M. Hugues, M. Teisseire, E. Beraudo, M. Jublot, P. Vennéguès, J. Zuniga-Pérez <i>CRHEA-CNRS, France ; Faculté des Sciences de Saint Jérôme – CP2M, France</i>	204
Tu-5p	Research of self-organization of iridium nano-islands for GaN nanowire growth <u>T. Grinys</u> , M. Šilinskas, A. Mekys, A. Kalpakovaitė, A. Kadys, T. Malinauskas and R. Tomašiūnas <i>Institute of Applied Research, Vilnius University, Lithuania; Institute of Micro- and Sensor Systems, Otto von Göricke University Magdeburg, Germany</i>	205
Tu-6p	Photoluminescence polarization in strained GaN/AlGaIn core/shell nanowire heterostructures <u>G. Jacopin</u> , P. Lavenus, L. Rigutti, S. Bellei, F. H. Julien, A.V. Davydov, D. Tsvetkov, K.A. Bertness and M. Tchernycheva <i>Institut d'Electronique Fondamentale UMR CNRS 8622, Université Paris Sud, France; MML, NIST, USA; PML, NIST, USA</i>	206

		page
Tu-7p	Electrical and luminescent properties of nanopillar GaN/InGaN MQW LED structures prepared from standard planar structures by dry etching using self-organized Ni nanomask <u>A.Y. Polyakov</u> , D.-W. Jeon, L.-W. Jang, I.-H. Lee, N.B. Smirnov, A.V. Govorkov, and E.B. Yakimov <i>Institute of Rare Metals, Russia ; School of Advanced Materials Engineering and Research Center for Advanced Materials Development, Chonbuk National University, Korea; Institute of Microelectronics Technology and High Purity Materials Russian Academy of Science, Russia</i>	207
Tu-8p	High quality epitaxial r-BN grown by CVD <u>M. Chubarov</u> , H. Pedersen, H. Högberg, A. Henry <i>Department of Physics, Chemistry and Biology, Linköping University, Sweden</i>	208
Tu-9p	Surface morphology of Al_{1-x}In_xN grown by low pressure MOVPE on various templates <u>E.R. Buß</u> , U. Rossow, H. Bremers and A. Hangleiter <i>Institute of Applied Physics, Technische Universität Braunschweig, Germany</i>	209
Tu-10p	Composition dependence of critical thicknesses in GaInN/GaN characterized by in situ X-ray diffraction measurement <u>Y. Kondo</u> , D. Iida, T. Sugiyama, M. Sowa, H. Matsubara, M. Iwaya, T. Takeuchi, S.Kamiyama, and I. Akasaki <i>Faculty of Science and Technology, Meijo University, Japan; Akasaki Research Center, Nagoya University, Japan</i>	210
Tu-11p	X-ray multiple diffraction as a new approach to structural characterization of III-nitride layers <u>R. Kyutt</u> , and M. Scheglov <i>Ioffe Physical Technical Institute, Russia</i>	211
Tu-12p	Structural characterization of short period superlattices GaN/AlN <u>R. Kyutt</u> , M. Scheglov, V. Ratnikov, V. Davydov, A. Smirnov, M. Yagovkina, M. Rojavskaja, E. Zavarin, and W. Lundin <i>Ioffe Physical Technical Institute, Russia</i>	212
Tu-13p	Detailed TEM quantifications of threading dislocations in GaN layers grown on Si <u>J. M. Manuel</u> , F. M. Morales, R. García, R. Aidam, L. Kirste, O. Ambacher <i>Dep. Ciencia de los Materiales e IM y QI., F. Ciencias, Universidad de Cádiz, Spain; Fraunhofer Institute for Applied Solid State Physics, Germany</i>	213

		page
Tu-14p	Comprehensive TEM quantification of threading dislocations scaling up through GaN and InN layers grown on sapphire <u>J. M. Mánuel</u> , D. Carvalho, R. Félix, F. M. Morales, T. Ben, R. García, A. Knübel, R. Aidam, L. Kirste, O. Ambacher, M. Himmerlich <i>Dep. Ciencia de los Materiales e IM y QI., F. Ciencias, Universidad de Cádiz, Spain; Fraunhofer Institute for Applied Solid State Physics,, Germany; Institut für Mikro- und Nanotechnologien, TU Ilmenau, Germany</i>	214
Tu-15p	Semipolar gallium nitride layers on Si grown by HVPE: TEM investigation L.M. Sorokin, A.E. Kalmykov, <u>A.V. Myasoedov</u> , V.N. Bessolov, A.V. Osipov and S. A. Kukushkin <i>Ioffe Physical Technical Institute, Russian Academy of Sciences, Russia; Institute for Problems of Mechanical Engineering, Russian Academy of Sciences, Russia</i>	215
Tu-16p	Comparison of the crystalline quality of GaN films on a- and c-sapphire substrates using low-temperature AlN buffer layers <u>K. Okuno</u> , T. Oshio, N. Shibata, S. Tanaka and H. Amano <i>Department of Electronics, Nagoya University, Japan; EcoTopia Science Institue, Nagoya University, Japan; Toyoda Gosei Co., Ltd., Optoelectronics Business Unit, Japan</i>	216
Tu-17p	Evaluation of Metastable-Phase Inclusion in PR-MOVPE-Grown InN Films by EBSD <u>T. Iwabuchi</u> , H. Matsumura, K. Prasertsuk, T. Kimura, Y. Zhang, Y. Liu, R. Katayama, and T. Matsuoka <i>Institute for Materials Research, Tohoku University, Japan; CREST, Japan Science and Technology Agency, Japan</i>	217
Tu-18p	Structural Study on Nitrated C-sapphire Substrate by Grazing Incidence X-ray Diffraction and Transmission Electron Microscopy <u>J.-S. Ha</u> , S.W. Lee, H.-J. Lee, S.-K. Hong, M.W. Cho, T. Yao <i>Faculty of Applied Chemical Engineering, Chonnam National University, Republic of Korea; Center for Interdisciplinary Research, Tohoku University, Japan; Materials Science and Engineering, Dong-A University, Republic of Korea; Department of Materials Science and Engineering, Chungnam National University, Republic of Korea</i>	218
Tu-19p	A Study on the Heteroepitaxy GaN Growth on Various Powders by Hydride Vapor Phase Epitaxy H.-J. Lee, S.W. Lee, <u>J.-S. Ha</u> , S.-K. Hong, M.W. Cho, T. Yao <i>Materials Science and Engineering, Dong-A University, Republic of Korea; Center for Interdisciplinary Research, Tohoku University, Japan; Faculty of Applied Chemical Engineering, Chonnam National University, Republic of Korea; Department of Materials Science and Engineering, Chungnam National University, Republic of Korea</i>	219

		page
Tu-20p	Annealing effect for unification of in-plane domain in AlN layers grown from Ga-Al flux <u>M. Adachi</u> , M. Sugiyama, A. Tanaka and H. Fukuyama <i>Institute of Multidisciplinary Research for Advanced Materials (IMRAM), Tohoku University, Japan; Sumitomo Metal Mining Co. Ltd., Japan</i>	220
Tu-21p	Nano-layers of nitride semiconductors grown by MEAGlow epitaxial technology and their low-dimensional optical properties <u>D. Alexandrov</u> , K.S. Butcher, P. Terziyska, R. Gergova, P. Binsted, D. Georgieva, V. Georgiev, B.Kemp <i>Lakehead University, Canada; Meaglow Ltd., Canada</i>	221
Tu-22p	Migration Enhanced Afterglow Growth of GaN at Low Temperatures <u>K.S.A. Butcher</u> , P. Terziyska, D. Alexandrov, V. Georgiev, D. Georgieva, P. W. Binsted, T. Menkad and R. Gergova <i>Semiconductor Research Laboratory, Department of Electrical Engineering, Lakehead University, Canada; Meaglow Ltd, Canada</i>	222
Tu-23p	Growth of thick AlN and GaN layers with atomically smooth and droplets-free surface by PA MBE under strongly metal rich conditions <u>P. Aseev</u> , A. Mizerov, D. Nechaev, V. Jmerik and S. Ivanov <i>Ioffe Physical Technical Institute, Russia</i>	223
Tu-24p	GaN electron spectra and lattice structural parameters under elastic strains <u>V.N. Brudnyi</u> , Kosobutsky, N.G. Kolin <i>National Research Tomsk State University, Russia; Kemerovo State University, Russia; L.Ya. Karpov Physical Chemical Institute, Russia</i>	224
Tu-25p	Homogeneous epitaxial InN films on the two-inch Al₂O₃ substrates grown by LP- MOCVD method <u>Yu. Buzynin</u> , A. Birukov, Yu. Drozdov, O. Khrykin, M. Drozdov, A. Lukyanov, E. Skoroxodov, V. Shashkin <i>Institute for Physics of Microstructures RAS, Russia; Scientific Research Physicotechnical Institute, Russia</i>	225
Tu-26p	The growth and characterization of In rich InGaN solar cell epitaxial structures by MOCVD <u>H. Çakmak</u> , M. Rudzinski, P. Demirel, A. Emen, W.Strupinski, and E. Özbay <i>Nanotechnology Research Center, Bilkent University, Turkey; Institute of Electronic Materials Technology Poland; Department of Physics, Bilkent University, Turkey; Department of Electrical and Electronics Engineering, Bilkent University, Turkey</i>	226

		page
Tu-27p	Influence of the V/III ratio on quality of thick epitaxial AlN layers grown on sapphire by High Temperature Hydride Vapor Phase Epitaxy (HT-HVPE) <u>N. Coudurier</u> , R. Boichot, A. Claudel, V. Fellmann, E. Blanquet, S. Coindeau, R. Martin, D. Pique, M. Pons <i>Science et Ingénierie des Matériaux et des Procédés, Grenoble INP-CNRS-UJF, France; ACERDE, France; CMTC, Grenoble INP-CNRS, France</i>	227
Tu-28p	Influence of the V/III ratio in the gas phase on thin epitaxial AlN layers grown on (0001) sapphire by High Temperature Hydride Vapor Phase Epitaxy (HT-HVPE) <u>N. Coudurier</u> , A. Claudel, V. Fellmann, M. Balaji, E. Blanquet, R. Boichot, S. Coindeau, B. Doisneau, R. Martin, S. Luca, A. Crisci, D. Pique, K. Baskar, M. Pons <i>ACERDE, France; Science et Ingénierie des Matériaux et des Procédés, Grenoble INP-CNRS-UJF, France; Anna University, India; CMTC, Grenoble INP-CNRS, France</i>	228
Tu-29p	Understanding and controlling In incorporation on InGaN surfaces: An ab initio approach <u>A. I. Duff</u> , L. Lymperakis, and J. Neugebauer <i>Max-Planck-Institut für Eisenforschung GmbH, Germany</i>	229
Tu-30p	HVPE of Al_{0.45}Ga_{0.55}N layers on trench patterned sapphire <u>S. Hagedorn</u> , E. Richter, U. Zeimer and M. Weyers <i>Ferdinand-Braun-Institut, Leibniz-Institut für Höchstfrequenztechnik, Germany</i>	230
Tu-31p	Top-side satellite to satellite temperature control in planetary MOCVD reactors using a novel gas-foil-rotation based temperature controller L. Pauli, D. Brien, M. Dauelsberg, M. Luenenbuerger, <u>F. Schulte</u> , B. Schineller, M. Heuken <i>AIXTRON SE, Germany</i>	231
Tu-32p	Thermodynamic analysis of InGaN-HVPE growth using III-bromides and III-iodides <u>T. Hirasaki</u> , K. Hanaoka, H. Murakami, Y. Kumagai and A. Koukitu <i>Department of Applied Chemistry, Tokyo University of Agriculture and Technology, Japan</i>	232
Tu-33p	GaN layers growth on pseudo (111)Al substrates by RF-MBE and their chemical lift-off technique <u>T. Honda</u> , M. Hayashi, Y. Sugiura, I. Takezawa and T. Yamaguchi <i>Department of Electrical Engineering and Electronics, Graduate School of Engineering, Kogakuin University, Japan</i>	233
Tu-34p	Strain relaxed growth of n-GaN epilayers M. Han, Y.S. Katharria1, J.H. Ryu, H.Y. Kim, H.K. Kim, <u>K.B. Ko</u> , N. Han, J.H. Kang, Y.J. Park, B.D. Ryu, E.-K. Suh, C.-H. Hong <i>Semiconductor Physics Research Center, School of Semiconductor and Chemical Engineering, Chonbuk National University, South Korea</i>	234

		page
Tu-35p	Growth and Characterization of the AlGaN epilayers grown on the variation of high temperature AlN buffer layer thickness by HVPE <u>J. Hwang</u> , J.S. Lee, H.-S. Kim, J.-H. Kim, T.-Y. Lim, H.-K. Oh, Y.J. Choi and H.-Y. Lee <i>Korea Institute of Ceramic Engineering & Technology, Korea; LumiGNtech Co., Korea</i>	235
Tu-36p	Laser patterning – an alternative method to prepare sapphire substrate for GaN growth <u>E. Jelமாகas</u> , A. Kadys, T. Malinauskas, T. Grinys, R. Tomašiūnas, P. Gečys, G. Račiukaitis <i>Vilnius University, Institute of Applied Research, Lithuania; Center for Physical Sciences and Technology, Lithuania</i>	236
Tu-37p	Stability of GaN(0001) surface coverage at mixed ammonia and hydrogen ambient <u>P. Kempisty</u> , P. Strąk, <u>S. Krukowski</u> <i>Institute of High Pressure Physics, Polish Academy of Sciences, Poland; Interdisciplinary Centre for Modelling, University of Warsaw, Poland</i>	237
Tu-38p	Hydrogen influence on adsorption of indium and gallium at GaN(0001) surface in MOVPE ambient – ab initio study <u>S. Krukowski</u> , P. Kempisty, P. Strąk, M. Leszczyński, R. Czernecki <i>Institute of High Pressure Physics, Polish Academy of Sciences, Poland; Interdisciplinary Centre for Modelling, University of Warsaw, Poland</i>	238
Tu-39p	Polarity inversion mechanism at oxidized AlN layers <u>E. Kishikawa</u> , K. Ueno, S. Inoue, J. Ohta, H. Fujioka, and M. Oshima <i>Department of Applied Chemistry, University of Tokyo, Japan; Institute of Industrial Science, University of Tokyo, Japan; Core Research for Evolutional Science and Technology, Japan Science and Technology Agency, Japan</i>	239
Tu-40p	Indium incorporation during growth of InGaN on (0001) surface <u>F. Krzyzewski</u> , M. Zaluska-Kotur <i>Institute of Physics, Polish Academy of Sciences, Poland</i>	240
Tu-41p	High-quality AlN layer deposited at high substrate temperature with high sputter power by RF reactive sputtering <u>T. Kumada</u> , M. Ohtsuka, and H. Fukuyama <i>Institute of Multidisciplinary Research for Advanced Materials, Tohoku University, Japan</i>	241

		page
Tu-42p	Spatial stress distribution of GaN grown on a serpentine masked structure L. Li, P.Liu, L. Liu, D. Li, W. Chen, Z. Yang, Y. Xie, <u>X. Hu</u> , and G. Zhang <i>State Key Laboratory for Artificial Microstructure and Mesoscopic Physics, School of Physics, Peking University, China; Department of Materials Science and Engineering, University of California Los Angeles, USA</i>	242
Tu-43p	Peculiarities of fast AlGa_N growth in planetary reactor <u>W.V. Lundin</u> , A.E. Nikolaev, M.M. Rozhavskaya, E.E.Zavarin, A.V. Sakharov, S.I. Troshkov, M.A. Yagovkina, and A.F. Tsatsulnikov <i>Ioffe Physical Technical Institute, Russia</i>	243
Tu-44p	Enhancement of In-incorporation into InGa_N by nitridation of sapphire substrate in MOVPE J. H. Choi, S. Kumar, K. Shojiki, T. Hanada, R. Katayama, and <u>T. Matsuoka</u> <i>Institute for Materials Research, Tohoku University, Japan; JST-CREST, Japan</i>	244
Tu-45p	High quality Ga_N layer grown on sapphire substrate using AlN/AlGa_N buffer for UV applications <u>A.V. Mazalov</u> , D.R. Sabitov, V.A. Kureshov, A.A. Padalitsa, A.A. Marmalyuk, R.Kh. Akchurin <i>Sigm Plus Co., Russia; Moscow University of Fine Chemical Technology, Russia</i>	245
Tu-46p	Effect of Dislocation Blocking on HVPE-grown AlN Using the Grooved Seed in Triangle-shaped Stripe <u>H. Miyake</u> , T. Nomura, and K. Hiramatsu <i>Department of Electrical and Electronic Engineering, Mie University, Japan</i>	246
Tu-47p	HVPE growth of a-plane (Al,Ga)_N layers on patterned c-plane sapphire <u>A. Mogilatenko</u> , S. Hagedorn, E. Richter, U. Zeimer, M. Weyers, G. Tränkle <i>Ferdinand-Braun-Institut, Leibniz-Institut fuer Hoehstfrequenztechnik, Berlin, Germany</i>	247
Tu-48p	Substrate bias effect in AlN buffer layers for InN films deposition by RF reactive sputtering <u>L. Monteagudo-Lerma</u> , S. Valdueza-Felip, M. González-Herráez, E. Monroy, F.B. Naranjo <i>Grupo de Ingeniería Fotónica, Departamento de Electrónica (EPS) Universidad de Alcalá, Spain; CEA-Grenoble, INAC/SP2M/NPSC, France</i>	248

		page
Tu-49p	Study of AlN layers grown on sapphire by RF reactive sputtering by a two-step deposition method <u>L. Monteagudo-Lerma</u> , S. Valdueza-Felip, M. González-Herráez, E. Monroy, F.B. Naranjo <i>Grupo de Ingeniería Fotónica, Departamento de Electrónica (EPS) Universidad de Alcalá, Spain; CEA-Grenoble, INAC/SP2M/NPSC, France</i>	249
Tu-50p	Temperature dependence of a-plane GaN low angle incidence microchannel epitaxy grown by ammonia-based metal-organic molecular beam epitaxy <u>S. Naritsuka</u> , C.H. Lin, S. Uchiyama, and T. Maruyama <i>Department of Materials Science and Engineering, Meijo University, Japan</i>	250
Tu-51p	Initial growth stages of AlN/c-Al₂O₃ by molecular beam epitaxy <u>D. Nechaev</u> , A. Mizerov, P. Aseev, Y. Kuznetsova, A. Sitnikova, V. Ratnikov, V. Jmerik, and S. Ivanov <i>Ioffe Physical-Technical Institute, Russia</i>	251
Tu-52p	Highly n-type conductive high Al-content Al_xGa_{1-x}N (x>0.7) grown by hot-wall MOCVD <u>D. Nilsson</u> , X.T. Trinh, U. Forsberg, N.T. Son, E. Janzén, and A.Kakanakova-Georgieva <i>Department of Physics, Chemistry and Biology, Linköping University, Sweden</i>	252
Tu-53p	Plasma-assisted electroepitaxy of GaN layers <u>S.V. Novikov</u> and C.T. Foxon <i>School of Physics and Astronomy, University of Nottingham, UK</i>	253
Tu-54p	Characterization of ultrathin InN films grown on YSZ substrates <u>K. Okubo</u> , A. Kobayashi, J. Ohta, M. Oshima and H. Fujioka <i>Institute of Industrial Science, University of Tokyo, Japan; Department of Applied Chemistry, University of Tokyo, Japan; CREST, Japan Science and Technology Agency, Japan</i>	254
Tu-55p	Pyrolysis of dimethylhydrazine for the MOVPE growth of GaN and InN monitored by in-situ quadrupole mass spectroscopy Q. T. Thieu, T. Inamoto, S. Kuboya, and <u>K. Onabe</u> <i>Department of Advanced Materials Science, University of Tokyo, Japan</i>	255
Tu-56p	Epitaxial growth of semipolar InAlN with high In concentrations on yttria-stabilized zirconia substrates <u>M. Oseki</u> , A. Kobayashi, J. Ohta, H. Fujioka and M. Oshima <i>Department of Applied Chemistry, University of Tokyo, Japan; Institute of Industrial Science, University of Tokyo, Japan; CREST, Japan Science and Technology Agency, Japan</i>	256

		page
Tu-57p	Growth of GaN layers with low dislocation density using specialized MBE system <u>S.I. Petrov</u> , A.N. Alexeev, D.M. Krasovitsky, V.P. Chaly, V.V. Mamaev <i>SemiTEq JSC, Russia; Svetlana-Rost JSC, Russia</i>	257
Tu-58p	Surface reconstruction affecting topography during metal-organic vapour phase epitaxy of nitrides <u>M. Pristovsek</u> , A. Kadir, K. Bellmann, F. Mehnke, J. Stellmach, C. Meissner, M. Leyer, T. Schwaner, T. Simoneit, M. Kneissl <i>Institut für Festkörperphysik, Technical University of Berlin, Germany</i>	258
Tu-59p	Selective area growth of GaN on r-plane sapphire by MOCVD <u>M.M. Rozhavsckaya</u> , W.V. Lundin, E.E. Zavarin, S.I. Troshkov, A.F. Tsatsulnikov <i>Ioffe Physical Technical Institute, Russia</i>	259
Tu-60p	Various types of GaN/InGaN nanostructures grown by MOCVD on Si(111) substrate <u>M.M. Rozhavsckaya</u> , W.V. Lundin, E.E. Zavarin, E.Yu. Lundina, S.I. Troshkov, V.Yu. Davydov, M.A. Yagovkina, P.N. Brunkov, A.F. Tsatsulnikov <i>Ioffe Physical Technical Institute, Russia</i>	260
Tu-61p	Routes to Aluminium Gallium Nitride Buffer Layers for UV-LED Growth <u>T.C. Sadler</u> , H.N. Li, V. Zubialevich, M. Conroy, Z. Quan, P.J. Parbrook <i>Tyndall National Institute, University College Cork, Ireland</i>	261
Tu-62p	Influence of Sapphire Substrate Miscut Angle on Indium Content of MOVPE-grown InGaN Films <u>K. Shojiki</u> , H. Shindo, S. Y. Ji, V. S. Kumar, J. H. Choi, Y. H. Liu, T. Hanada, R. Katayama, and T. Matsuoka <i>Institute for Materials Research, Tohoku University, Japan; CREST, Japan Science and Technology Agency, Japan</i>	262
Tu-63p	Growth of GaN layer with high crystallinity on free-standing GaN substrate using Ga₂O as Ga source <u>T. Sumi</u> , J. Takino, Y. Bu, A. Kitamoto, M. Imade, M. Yoshimura, M. Isemura, Y. Mori <i>Osaka University, Japan; Itochu Plastics Inc., Japan</i>	263

		page
Tu-64p	Effect of Oxygen Partial Pressure on the Growth of Single-Crystalline Aluminum Nitride Layer using Liquid Phase Epitaxy Technique <u>M. Takasugi</u> , M. Adachi, M. Sugiyama, A. Tanaka, and H. Fukuyama <i>Institute of Multidisciplinary Research for Advanced Materials (IMRAM), Tohoku University, Japan; Sumitomo Metal Mining Co., Ltd., Japan</i>	264
Tu-65p	Dramatic improvement in the crystalline quality of AlN grown on thermally-nitrided sapphire substrates by pulsed sputtering deposition <u>K.Ueno</u> , E. Kishikawa, S. Inoue, J. Ohta, H. Fujioka, M. Oshima and H. Fukuyama <i>Department of Applied Chemistry, University of Tokyo, Japan; Institute of Industrial Science, University of Tokyo, Japan; Core Research for Evolutional Science and Technology, Japan Science and Technology Agency, Japan; Institute of Multidisciplinary Research for Advanced Materials, Tohoku University, Japan</i>	265
Tu-66p	A study of GaN regrowth on the micro-faceted GaN template formed by in-situ HCl etching W. Luo, <u>J. Wu</u> , X. Liu, X. Kang, T. Yu and G. Zhang <i>Research Center for Wide-gap Semiconductors, School of Physics, Peking University, P. R. China</i>	266
Tu-67p	Step bunching and meandering induced by natural step flow and its role in the dynamics of crystal growth <u>M.A. Załuska-Kotur</u> , F. Krzyżewski, and S. Krukowski <i>Institute of Physics, Polish Academy of Sciences, Poland; Faculty of Mathematics and Natural Sciences, Card. Stefan Wyszyński University, Poland; High Pressure Research Center, Polish Academy of Sciences, Poland and Interdisciplinary Centre for Materials Modeling, Warsaw University, Poland</i>	267
Tu-68p	Growth of AlGa_N on sapphire using AlN/GaN superlattices as strain relaxing layers <u>V. Zubialevich</u> , T. Sadler, H. Li, Z. Quan, P. Parbrook <i>Tyndall National Institute, University College Cork, Ireland</i>	268
Tu-69p	AlGa_N/AlN quantum dots for UV light emitters <u>C. Himwas</u> , M. den Hertog, F. Donatini, Le Si Dang, L. Rapenne, E. Sarigiannidou, R. Songmuang, and E. Monroy <i>CEA-CNRS Group “Nanophysique et Semiconducteurs”, Institut Néel-CNRS, France; INP-Grenoble/Minatec, France; CEA-CNRS Group “Nanophysique et semiconducteurs”, INAC-SP2M, CEA-Grenoble, France</i>	269

		page
Tu-70p	Growth of InN Nanostructures by Droplet Epitaxy Techniques <u>M. Kumar</u> , T.N Bhat, B. Roul, M.K Rajpalke, A.T. Kalghatgi and S.B. Krupanidhi <i>Materials Research Centre, Indian Institute of Science, India; Central Research Laboratory, Bharat Electronics, India</i>	270
Tu-71p	UV Photoresponse of GaN Nanodots grown by Molecular Beam Epitaxy <u>M. Kumar</u> , B. Roul, M.K. Rajpalke, T.N. Bhat, A.T. Kalghatgi and S.B. Krupanidhi <i>Materials Research Centre, Indian Institute of Science, India; Central Research Laboratory, Bharat Electronics, India</i>	271
Tu-72p	Patterning of Pd and AlGaIn/GaN superlattice for the fabrication of blue laser diodes J.-K. Kim, D.-M. Kim, S.-J. Hwang, M.-J. Park, S.-N. Lee, and <u>J.-M. Lee</u> <i>Department of Materials Science and Metallurgical Engineering, Suncheon National University, Korea; Department of Nano-Optical Engineering, Korea Polytechnic University, Korea</i>	272
Tu-73p	InGaIn quantum dot formation mechanism on hexagonal GaN/InGaIn/GaN pyramids <u>A. Lundskog</u> , J. Palisaitis, C.W. Hsu, M. Eriksson, K. F. Karlsson, L. Hultman, P.O.Å. Persson, U. Forsberg, P.O. Holtz, E. Janzén <i>Department of Physics, Chemistry and Biology (IFM), Linköping University, Sweden</i>	273
Tu-74p	Controlled growth of hexagonal GaN pyramids by hot-wall MOCVD <u>A. Lundskog</u> , Chih-Wei Hsu, U. Forsberg, P.O. Holtz, E. Janzén <i>Department of Physics, Chemistry and Biology (IFM), Linköping University, Sweden</i>	274
Tu-75p	InGaIn light-emitting diodes with the complex air structures with combination of triangular prisms and cones <u>V. Lysak</u> , J.H. Kang, and C.H. Hong <i>Chonbuk National University, South Korea</i>	275
Tu-76p	Optical Polarization Properties of a-plane GaN Light Emitting Diodes by Controlled Integration of Silica Nano-spheres <u>S.H. Park</u> , J. Jang, D. Moon, D.-U. Kim, H. Chang, H. Jeon, J. Xu, Y. Nanishi, and E. Yoon <i>Department of Materials Science and Engineering, Seoul National University, Korea; Hybrid Materials Program, Department of Materials Science and Engineering, Seoul National University, Korea; Department of Physics and Astronomy, Seoul National University, Korea; Department of Biophysics and Chemical Biology, Seoul National University, Korea; Department of Engineering and Physics, Brown University, USA; Department of Photonics, Ritsumeikan University, Japan; Department of Nano Science and Technology, Seoul National University, Korea</i>	276

		page
Tu-77p	Layer thickness dependent electrical properties of MOCVD grown GaN layers A. Kadys, S. Nargelas, <u>T. Malinauskas</u> , S. Lapinskas, A. Arnatkevičiūtė, T. Grinys, R. Tomašiūnas, and K. Jarašiūnas <i>Institute of Applied Research, Vilnius University, Lithuania</i>	277
Tu-78p	Resistance formation mechanisms for contacts to trinitride heterostructures with high dislocation density A.V. Sachenko, A.E. Belyaev, N.S. Boltovets, Yu.V. Zhilyaev, V.P. Klad'ko, <u>R.V. Konakova</u> , Ya.Ya. Kudryk, V.N. Panteleev, V.N. Sheremet <i>V. Lashkaryov Institute of Semiconductor Physics, NAS of Ukraine Ukraine; State Enterprise Research Institute "Orion", Ukraine; Ioffe Physico-Technical Institute, Russia</i>	278
Tu-79p	Ohmic and Schottky contacts to AlGaIn layers with high Al content for deep ultraviolet optoelectronics <u>S. Tarasov</u> , I. Lamkin, E. Menkovich, T. Komissarova, V. Jmerik, S. Ivanov <i>Saint Petersburg Electrotechnical University "LETI", Russia; Ioffe Physical-Technical Institute, Russia</i>	279
Tu-80p	Drastic change in electronic structure of AlGaIn under Ba adsorption <u>S. Timoshnev</u> , G. Benemanskaya, S. Ivanov, V. Jmerik, and M. Lapushkin <i>Ioffe Physical Technical Institute, Russia</i>	280
Tu-81p	Electrochemical profiling with verifying by atomic force microscopy of heterostructures with multiple quantum wells InGaIn/GaN <u>V. Zubkov</u> , O. Kucherova, D. Frolov and A. Zubkova <i>St. Petersburg State Electrotechnical University "LETI", Russia</i>	281
Tu-82p	Ultrashort laser pulses GaN ablation mechanisms I.N. Zavestovskaya, P.G. Eliseev, O.N. Krokhin, <u>N.A. Kozlovskaya</u> <i>P.N. Lebedev Physical Institute of Russian Academy of Science, Russia; Center for High Technology Materials, UNM, USA</i>	282
Tu-83p	On the study of growth of InN by PAMBE <u>R. Yang</u> , T. Krzyzewski and T. Jones <i>University of Warwick, UK</i>	283
Tu-84p	Effects of Crystallography-etched Facets on Patterned Sapphire Substrates for GaN-based LEDs <u>R.-H. Horng</u> , K.-C. Shen, H.-H. Hsueh, and D.-S. Wu <i>Graduate Institute of Precision Engineering, National Chung Hsing University, Taiwan, ROC; Department of Materials Science and Engineering, National Chung Hsing University, Taiwan, ROC; Department of Electrical Engineering, Da-yeh University, Taiwan, ROC</i>	284

		page
Tu-85p	Improving the internal quantum efficiency of GaN by removing the dislocations <u>Y.-H. Yeh</u> , C.-M. Chu, C.-H. Lee, Y.-H. Wu, Y.-Ch. Hsu, T.-Y. Yu, W.-I Lee <i>Department of Electrophysics, National Chiao Tung University, Taiwan</i>	285
Tu-86p	A-plane GaN thick films grown by hydride vapor phase epitaxy <u>Y.-H. Wu</u> , C.-H. Lee, C.-M. Chu, Y.-H. Yeh, C.-L. Chen and W.-I Lee <i>Department of Electrophysics, National Chiao Tung University, Taiwan</i>	286
Tu-87p	Single-wire photodetectors and light emitting diodes based on GaN/InGaN radial nanowire heterostructures <u>P.Lavenus</u> , G.Jacopin, A.De Luna Bugallo, L.Rigutti, F.H.Julien, C.Durand, D.Salomon, J.Eymery, L.F.Zagonel, M.Kociak and M.Tchernycheva <i>Institut d'Electronique Fondamentale UMR CNRS 8622, Univ. Paris Sud 11, France; Equipe mixte CEA-CNRS "Nanophysique et semiconducteurs", SP2M, UMR-E CEA/UJF-Grenoble 1, INAC, France; CEA-Leti, MINATEC campus, France; Laboratoire de Physique des Solides, UMR CNRS 8502, Univ. Paris Sud 11, France</i>	287
Tu-88p	Optical properties of the InGaN light-emitting diodes grown on different-shaped pattern sapphire substrates <u>C.-F. Lin</u> , C.-H. Hsieh, C.-M. Lin, J.-J. Dai and C.-C. Yang <i>Department of Materials Science and Engineering, National Chung Hsing University, Taiwan</i>	288

Abstracts

Review on growth, properties, and applications of AlInN

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Contrary to III-V arsenides for which GaAs/AlGaAs heterostructures can be fabricated with nearly lattice-matched condition for the whole Al composition range, group-III nitride semiconductors possess markedly different lattice constant, which might be detrimental for many applications. For instance, AlGaN/GaN two-dimensional electron gas (2DEG) heterostructures may undergo strain relaxation for an Al content exceeding 40%, which limits in some extend their application to high power electronics. On the other hand, optical confinement involving AlGaN layers becomes rapidly an issue when increasing the Al content, although the refractive index contrast remains rather limited.

Then an alternative solution consists in using a wide bandgap III-N ternary alloy, namely InAlN, which can be lattice-matched to GaN for an In content of 17%. Such an alloy exhibits a bandgap around 4.4 eV, which corresponds to that of $\text{Al}_{0.46}\text{Ga}_{0.54}\text{N}$.

In this review, I will first present the growth of InAlN and discuss the structural and optical properties of this peculiar material. N and p type doping will then be addressed. We will see that a lattice-matched light-emitting diode emitting in the UV range can be fabricated on GaN substrate. This demonstrates that true p-type doping can be achieved, as also confirmed by C-V measurements. Finally, potential applications of InAlN in optoelectronics and electronics will be reviewed.

Future Prospect of Large Area Nitride Devices Prepared by Pulsed Sputtering Deposition

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We have developed a new epitaxial growth technique named PSD (pulsed sputtering deposition), which allows us to obtain high quality group III nitrides even at room temperature with high productivity. Various nitride devices such as LEDs and HEMTs have been already fabricated successfully by the use of PSD. In this presentation, we will discuss feasibility of large area nitride devices fabricated with the low temperature PSD technique on various low cost large area substrates such as metals or mica. We will also show that PSD is quite promising for fabrication of long wavelength light emitting devices and solar cells that utilize high In concentration InGaN.

It is generally believed that group III nitride devices exhibit high performance but they are quite expensive because their fabrication process involves low throughput high temperature epitaxial growth by MOCVD or MBE. It is quite natural to believe that group III nitride devices prevail quickly among various new application fields once low cost fabrication process is established. Large area nitride devices such as solar cells and displays are among these applications. For this purpose, we have recently developed a new growth technique named PSD (pulsed sputtering deposition). PSD has already attracted much attention of industry engineers because its productivity is much higher than that of MOCVD. It is known that various nitride devices such as LEDs and HEMTs can be fabricated by the use of PSD and operated successfully. In this technique, surface migration of the film precursors is enhanced and, therefore, the temperature for epitaxial growth is dramatically reduced. This reduction allows us to utilize various large area low cost substrates such as metal sheets or mica sheets that have not been used for growth of semiconductors so far due to their chemical vulnerability. In this presentation, we will discuss feasibility of large area nitride devices such as LED displays or solar cells fabricated with PSD on various low cost substrates. We will also show that PSD is quite promising for growth of high In concentration InGaN which is necessary for fabrication long wavelength optical devices.

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Semipolar nitride hetero-structures on patterned substrates

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Green light emitting diodes based on GaN and their alloys still suffer from fairly low performance as compared to shorter wavelength “blue” emitters. For this so-called “green gap”, two main reasons have been identified: i) The epitaxial growth of GaInN with high indium content typically results in low material quality owing to a large lattice mismatch to the GaN base material and limited miscibility of GaN and InN when forming a ternary alloy. ii) The lattice mismatch induced strain additionally causes the formation of huge piezoelectric fields within the GaInN quantum wells separating electrons and holes locally and hence reducing their recombination probability. By changing the main epitaxial growth direction of nitride heterostructures from the conventional polar c-direction into less polar crystal directions, the internal fields can be strongly reduced. They even vanish for growth along the non-polar m- and a-directions. Very impressive results have been obtained recently by growing longer wavelength device structures on semipolar GaN bulk substrates which have been prepared by cutting from several mm-thick c-plane wafers. However, owing to the small thickness of such wafers, the size of these substrates is limited to a few square mm preventing their use for mass production etc. On the other hand, hetero-epitaxial approaches on foreign substrates like sapphire or SiC of adequate orientation typically suffer from huge defect densities and in particular stacking faults, obviously a consequence of the deviation of the main growth direction from the well-established and most suitable c-direction.

Therefore, we are currently investigating several hetero-epitaxial approaches which may provide large areas of high quality semipolar nitride structures grown on easily available sapphire wafers. In the approach presented here, we use the c-direction, for which optimal growth parameters are best established, as main growth direction for eventually obtaining a flat semipolar GaN surface. In order to establish a {10-11} semipolar plane, n-plane sapphire having a {11-23} surface is chosen as substrate, as the c-direction of this substrate is nearly parallel to the c-direction of the final semipolar {10-11} GaN layer [1]. Striped grooves are etched into the sapphire surface by Reactive Ion Etching (RIE), the c-plane-like side walls of which act as nucleation areas for the subsequent GaN growth starting in c-direction. After coalescence of these stripes, closed {10-11} planes with fairly low stacking fault densities can be achieved, as confirmed by excellent photoluminescence spectra. They exhibit very flat surfaces with an RMS roughness of about 1.1 nm as measured by AFM; the original stripe structure is not reflected in the surface morphology. Similarly, {11-22} planes can be realized on r-plane sapphire [2]. In order to optimize the quality of the semipolar GaN layers and to accommodate the slight misorientation between the c-directions of the sapphire wafer and the semipolar GaN layer, we studied the influence of a slight miscut of the sapphire wafers. Indeed, x-ray data confirmed an improvement of the layer quality, although the shape of the etched grooves also changed significantly as a function of the miscut angle. GaInN quantum wells grown on such planar semipolar layers show homogeneous properties with intense and fairly narrow photoluminescence (FWHM of 200 meV at 465 nm). Moreover, the GaN layer quality could be further improved by overgrowth with a thicker GaN layer deposited by hydride vapor phase epitaxy.

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AlN-based technology for deep UV and high-power applications

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For the first time in history of III-nitrides, the availability of low defect density ($<10^3 \text{ cm}^{-2}$) native AlN substrates offers an opportunity for growth of AlGaN alloys and device layers that exhibit million-fold lower defect densities than the incumbent technologies and enable one to assess and control optical and electrical properties in absence of extended defects. Epi-ready AlN wafers are fabricated from AlN boules grown by physical vapor transport at temperatures between 2200 and 2300°C. Gradual crystal expansion is achieved through a scalable, iterative re-growth process in which the high crystal quality is maintained over many generations of boules.

Despite the excellent crystal quality, below bandgap optical absorption bands in the blue/UV range affect the UV transparency of wafers. We use density functional theory (DFT) to develop a model to understand the interplay of point defects responsible for this absorption. We show a direct dependence of the mid-gap absorption band with the carbon concentration within the AlN.

Low defect density AlN and AlGaN epitaxial films are grown upon these wafers that exhibit superior optical properties in terms of emission efficiency and line width and can be doped with an efficiency that is several orders of magnitude higher than possible in technologies using non-native substrates. UV LED structures and Schottky diodes were fabricated on these materials that exhibit low turn-on voltages and breakdown fields greater than 10 MV/cm.

This presentation will review state-of-the-art of AlN-based technology and give examples of potential applications in future devices and contrast these with other wide bandgap technologies.

UV-C LEDs on free-standing AlN substrates

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The development of wide bandgap semiconductors over the past two decades has shown significant progress. Different Oxide-, Carbide-, and Nitride-based semiconductors have proven to be vital in broad spectrum of electronic devices. The nitrides continue to attract scientific interest due to their ability to form solid solutions with given material properties thus providing a wide range of applications. The aluminum nitride (AlN) and AlGaN-based semiconductor system plays key role in the ultraviolet (UV) optoelectronics as it naturally supplies coverage for light emission and detection for UV-C (germicidal) radiation range.

The mid-UV radiation, with wavelengths in the range 250 – 275 nm, effectively inhibits the ability of micro-organisms (e.g. viruses, bacteria) to reproduce. The UV-C light-emitting diodes (LEDs) have several distinct advantages compared to mercury-lamp based systems. These include small size, instant turn-on ability, ruggedness, lifetime, and safety. However, early approaches to UV-C LEDs were to grow the required $\text{Al}_x\text{Ga}_{1-x}\text{N}$ alloys on sapphire substrates and attempt to reduce the high threading dislocation density in the films (due both to large lattice and thermal-expansion mismatch) through novel nucleation and superlattice approaches. At best, these approaches have produced active regions for UV-C LEDs with threading dislocation densities in the range of 10^8 cm^{-2} .

At Crystal IS, we have, for a number of years now, followed the approach of growing high quality bulk crystals of AlN at high temperatures by physical vapor transport (PVT) with an optimized growth regime at higher than stoichiometric nitrogen pressures. It is also essential to prepare the surface of the substrates yielded from slicing these ingots. Particular attention has to be focused on the removal of subsurface damage. While high quality optical surfaces can be prepared with mechanical polishing, the subsurface damage introduced by such a technique prevents high quality pseudomorphic epitaxial growth. For Al-polarity, c-face orientation, we have demonstrated very high quality pseudomorphic $\text{Al}_x\text{Ga}_{1-x}\text{N}$ layers with low dislocation density, smooth surfaces, and high conductivity. Since no misfit dislocations are generated during pseudomorphic growth, no new threading dislocations are generated and it is therefore possible to grow thicker layers with a threading dislocation density comparable to the starting substrate.

From transmission electron microscopy and cathodoluminescence (CL) measurements, it is confirmed that the dislocation density in the n-type, $\text{Al}_{0.7}\text{Ga}_{0.3}\text{N}$ layer is low ($<10^5 \text{ cm}^{-2}$). The low dislocation density continues through the multi-quantum wells (MQW) and the electron blocking layer (EBL). The threading dislocations do not appear to propagate into the active region and thus enable the achievement of over 50% internal quantum efficiencies (IQE) from the devices fabricated on the low dislocation density pseudomorphic active layers. These layers are fabricated into mid-ultraviolet light emitting diodes with peak wavelengths in the range of 250-270 nm. Diodes have recently been demonstrated with greater than 35mW cw power at 260 nm (with 3.5% wall plug efficiency under these operating conditions) and greater than 200 mW pulsed power. These devices also have long lifetimes ($>10,000\text{h}$) at high currents ($>100 \text{ mA}$) which, again, is believed to be a benefit of the low defect densities in the active $\text{Al}_x\text{Ga}_{1-x}\text{N}$ layers.

Efficient current injection in 290 nm to 350 nm UV light emitting diodes

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Ultraviolet light emitting diodes (UV LED) are of great interest for a number of applications, e.g. UV curing, sensing, and phototherapy. LEDs with high external quantum efficiencies (EQE) still remain a great challenge. The heterostructure design of the (In)AlGa_N multiple quantum well (MQW) active region and the electron blocking layer (EBL) in the UV LED is crucial for the EQE and the emission characteristics. One problem is electron leakage from the MQW active region into the p-side of the LED, which reduces the efficiency and results in long wavelength defect luminescence. In addition, the EQE decreases with increasing aluminum content in the active region. This is attributed to reduced internal quantum efficiency (IQE) as well as reduced injection efficiency.

In order to separate these effects, we have studied the influence of the AlGa_N EBL barrier height on the injection efficiency of UV LEDs from 290 nm to 350 nm by measuring the EQE and IQE independently. The LEDs were grown by metal-organic vapor phase epitaxy (MOVPE) on 4 μm thick relaxed (0001) Al_{0.5}Ga_{0.5}N:Si layers with a threading dislocation density of $1 \times 10^{10} \text{ cm}^{-2}$. The results were compared with 1D drift diffusion simulations assuming a band offset of 70% in the conduction band and 30% in the valence band, respectively [1].

In a first series the AlGa_N EBL was kept constant at 60% aluminum content, resulting in different effective barrier heights by changing emission wavelength of the active region. The measurements show a strong increase of the injection efficiency when the wavelength is increased. The integrated output power at 100 mA increases from 0.3 mW at 290 nm to 1.2 mW at 305 nm. Already at 300 nm the output power is enhanced and p-side luminescence is strongly suppressed. This is due to increased barrier height in the conduction band between quantum well barriers and EBL. The barrier height increases from 260 meV for the 290 nm LED with Al_{0.6}Ga_{0.4}N EBL to 360 meV for 300 nm LED and 420 meV for the 305 nm LED, i.e. a minimum band offset of 360 meV is required to reduce the electron leakage currents.

The effective barrier height of the EBL can also be increased by shifting the Fermi energy within the EBL towards the valence band by increased p-doping. Even for designs with very large band offsets low p-doping leads to strong defect luminescence. In a second series the magnesium doping in the EBL was varied for 290 nm LEDs with an aluminum content of 70% in the EBL. Poor injection efficiency was found for very low and very high magnesium contents. The injection efficiency was increased and the defect luminescence was suppressed by optimized p-doping of the EBL.

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Improvement of light extraction efficiency of AlGaIn deep-UV LED using 2-dimensional photonic crystal (2D-PhC)

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Deep-ultraviolet (DUV) light-emitting diodes (LEDs) have a wide range of potential applications such as sterilization, water purification, medicine and biochemistry, white light illumination, and so on. However, the efficiency of AlGaIn DUV LED is not yet so high compared with blue LEDs due to inferior light extraction efficiency (LEE). In this study, we increased external quantum efficiency (EQE) of DUV-LEDs by improving the LEE using 2-dimensional photonic crystal structures (2D-PhC).

Figure 1 shows a schematic structure of a 270 nm-band DUV-LED using 2D-PhC. The 2D-PhC patterns with high aspect ratio were fabricated on the backside of c-plane sapphire substrates using the combination of nanoimprint lithography technique and ICP dry etching. We used air hole arrays in triangular lattice for the 2D-PhC. The air hole arrays are 250 nm in diameter and 120-200 nm in depth with the period of 300 nm. The layer structure of the sample is consisting of an approximately 4 μm -thick AlN layer and a 1.5 μm -thick Si-doped AlGaIn layer, an Si-doped AlGaIn buffer layer, an AlGaIn/AlGaIn multi (M)QW emitting layer, a Mg-doped AlGaIn/AlGaIn multi-quantum-barrier (MQB) electron-blocking layer (EBL), a Mg-doped AlGaIn layer and a Mg-doped GaN contact layer grown on a sapphire substrate with 2D-PhC on the backside. Figure 2 shows the comparison of EQE and electroluminescence (EL) spectra of DUV-LEDs between the samples with and without 2d-PhC patterns. EQE and EL spectra of AlGaIn DUV-LEDs were measured under room temperature (RT) CW operation. We obtained about 20% higher EQE for the 270 nm AlGaIn LED due to the increase of LEE by using 2D-PhC. It is predicted by the simulation calculation using FDTD method that the LEE is increased by maximally more than 1.5 times by optimizing the PhC structure.

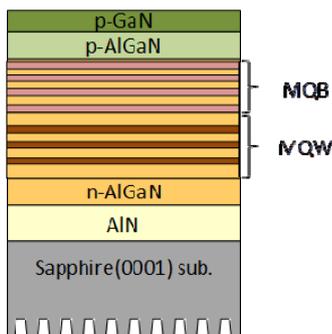


Fig. 1. Schematic structure of AlGaIn DUV-LED with 2D-PhC on the backside of substrate

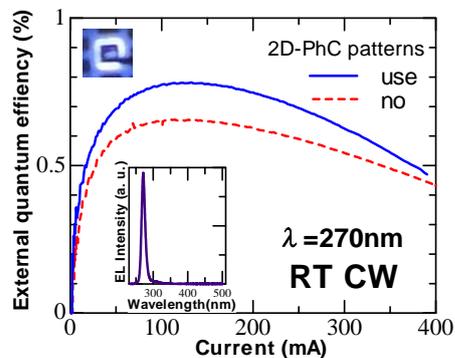


Fig.2. Comparison of EQE characteristics and EL spectra of DUV-LEDs between samples with and without 2D-PhC patterns

AlGaN MSM photodetectors for the UV-C spectral region on planar and ELO AlN/sapphire templates

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Photodetectors for the UV spectral region are required to control high-power UV lamps, UV LEDs and excimer lasers in UV lithography, disinfection, and medical applications. Due to their direct bandgap, group III-nitrides are highly promising for UV detectors that are visible- or solar blind and very resistant against radiative damage. Their spectral response can be tuned by absorber material composition and incorporation of heterojunctions. Many operating figures of such AlGaN photodetector like responsivity, dark current or response time strongly depend on the crystal quality of the absorber layer. In this paper solar-blind metal-semiconductor-metal photodetectors (MSM-PD) with different dislocation densities are discussed.

For UV-C photodetectors 1.4 μm thick non-intentionally doped $\text{Al}_x\text{Ga}_{1-x}\text{N}$ absorber layers with $x = 0.4$ were deposited by MOVPE on sapphire substrates with two kinds of AlN buffer layers. Type A has a planar 500 nm thick AlN layer with a screw dislocation density of 10^8 cm^{-2} and an edge dislocation density of 10^{10} cm^{-2} . Type B is an epitaxial lateral overgrown (ELO) template with the same screw dislocation density, but one order of magnitude smaller edge dislocation density. The stripe pattern of the type B template is oriented along the m-direction ($\langle\langle -1100 \rangle\rangle$) [1]. For thick $\text{Al}_{0.4}\text{Ga}_{0.6}\text{N}$ layers on type A template the dislocation density remains unchanged, whereas on type B the edge dislocation density is nearly twice with respect to the underlying ELO template. But the edge dislocation density of an $\text{Al}_{0.4}\text{Ga}_{0.6}\text{N}$ layer on type B is about four times lower than on type A templates. The MSM devices have two interdigitated Pt Schottky contacts with titanium and gold enforcement, 400 μm finger lengths, 2 μm finger width and various finger spacings.

MSM photodetectors on type A template were analyzed in dependence on the finger spacing. For a fixed bias the responsivity increases with decreasing electrode distance, which is caused by a short carrier lifetime compared to the transit time and a low carrier mobility in such highly Al containing AlGaN absorber layers. Nevertheless, the responsivity of the MSM-detector with 2 μm finger distance is as high as 145 mA/W at the spectral peak wavelength of 275 nm and a bias voltage of 5 V commonly used for MSM photodetectors. The responsivity spectrum shows a broad plateau for the desired spectral range from 240 nm to 280 nm. This detector is solar blind and the ratio between the responsivity at 275 nm and visible light (above 400 nm) is higher than 10^4 . The corresponding dark current is below 3 pA. The photocurrent depends linearly on the incident power density and thus the responsivity is independent on the incident power. MSM photodetectors on type B templates with lower edge dislocation density show about five times higher responsivity at 275 nm due to internal gain. However, this responsivity behaves sublinear with the irradiation level. A maximum absolute responsivity maximum of about 1 A/W is obtained near 240 nm and thus well below the cut-off wavelength expected for $\text{Al}_{0.4}\text{Ga}_{0.6}\text{N}$ based MSM-PDs. The dynamic behavior and the dark current will be discussed.

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Growth of bulk GaN by ammonothermal technique

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Gallium Nitride (GaN) is a very interesting material because of its applications in fabrication of optoelectronic devices operating in short wavelength region (blue and green light). Low dislocation bulk GaN seems to be good candidate for high-power laser optical systems. For example, GaN-based green light emitters are supposed to be more efficient and more compact than currently available second harmonic generation lasers. These lasers are desired to fill the green-gap, which is necessary for commercialization of red-blue-green (RGB) laser displays and pocket laser projectors of high pixel resolution.

This communication presents the properties of truly bulk GaN monocrystals, obtained by unique ammonothermal method (in so-called ammonobasic regime). The ammonothermal technology is regarded as an analogue of hydrothermal one, commonly used in industrial quartz production. In this method GaN feedstock is dissolved in supercritical ammonia in one zone of high pressure autoclave, then transported to another one via convection, where crystallization on GaN seeds takes place due to supersaturation of the solution. The crystal growth proceeds in temperature range $T=500-600^{\circ}\text{C}$ and pressure $p=0.1-0.3\text{ GPa}$. GaN produced this way possesses the crystal features such as exceptionally low FWHM value of X-ray rocking curve (20 arcsec), large lattice curvature radius ($R\sim 100\text{ m}$) and the lowest dislocation density (of the order of 10^3 cm^{-2}). The substrates of various crystallographic orientation – polar C-plane, semipolar and nonpolar M-plane can be machined. The two latter ones, due to the largely reduced electric fields, are the response of company to market demands for this type substrates for green optoelectronics purposes. The dimensions of commercially available substrates reach 2 inch in case of polar substrates and about 26mm x 26mm in case of non-polar and semi-polar ones, being the largest size of non- and semi-polar truly bulk material. The polishing progress results in epi-ready surface with clear visible atomic steps and RMS factor of the order of few Å in substrates of any orientation. The results of improved purity and transparency of ammonothermal GaN crystals will also be shown.

Finally, the examples of devices grown on ammonothermal substrates will be presented: light emitting diodes (LEDs), high-power laser diodes of outstanding parameters, proving their potential in blue and green optoelectronics. Moreover, possibility of large size (26mm x 26 mm) non- and semi-polar GaN substrates opens perspective of more efficient green laser production.

Specific features of sublimation growth of bulk AlN crystals on SiC wafers

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AlN crystals are of interest in such application as wafers for optoelectronic devices, among them ultraviolet light emitting diodes. The given report informs about some specific problems of growth of AlN crystals on SiC seeds by the sublimation method.

It is noted that the main problems of sublimation growth of AlN, including insufficient repeatability of results, are due to the fact that in the vapor phase there are very few combinations of Al and N, which can function as a transporter, and equilibrium vapor above AlN consists only of extremely reactive aluminum vapor and extremely stable in terms of energy nitrogen molecules. High reactivity of Al vapor, as well as Si, combined with high growth temperature extremely limits the number of materials suitable for container manufacture. The materials practically used for AlN growth, among them graphite, W, BN and TaC, can not be considered as fully inert, since they interact with Al and Si and contaminate the growth surface.

The report gives results of comparative study of growth of AlN on SiC seeds in containers made of preliminary carbidized Ta or tantalum carbide. It is shown that in case of use of Ta containers, preliminary carbidized by high-temperature annealing in graphite, the quality and repeatability of AlN growth highly depend on conditions of temperature annealing of the containers. At medium temperatures of the annealing, when in the process of annealing a TaC layer is formed on the Ta surface and phases of variable composition of Ta_mC_n are formed inside, high growth quality is possible only until cracks are formed in the crucible. The degradation of the growth quality is associated with contamination of the growth surface with carbon and tantalum impurities. At higher temperatures of the annealing it is possible to transform Ta to TaC, however, these containers are very fragile and short-lived. Alternatively we used TaC containers, made by pressing and further thermal processing of TaC powder.

The stability of molecular nitrogen conditions low condensation coefficient by sublimation growth of AlN. It is observed that the value of the local condensation coefficient at various areas of the surface strongly differs, that leads to high dependence of the growth rate on the surface condition and structural defect occurrence. The condensation coefficient generally decreases near structural defects, that leads to occurrence of voids and micropipes on the growing crystal surface. The abnormal condensation mechanism is caused by occurrence on the growing surface of a liquid phase, including aluminum and silicon.

The denoted problems highly influence the repeatability of the process and the crystal quality. However, the implemented work on optimization of conditions of AlN crystallization gave a possibility to provide a controlled growth of very qualitative bulk AlN crystals up to two inches diameter and up to 5 mm thick.

Growth of cm-order bulk GaN single crystals by Na-flux method with a necking technique

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A commercial availability of dislocation-free bulk GaN single crystals is the key breakthrough for the realization of the full potential of the GaN-based devices. The Na-flux method is a promising candidate for the mass production of high-quality GaN crystals because a decrease in dislocation density from 10^8 cm^{-2} in a seed to 10^4 cm^{-2} in the liquid-phase epitaxy (LPE) layers and a growth rate over $20 \mu\text{m/h}$ were attained with 2-inch GaN LPE. However, further breakthrough is needed to realize dislocation-free GaN single crystals. To overcome this big challenge, we focused attention on the necking technique in the Czochralski (CZ) growth of Si ingot. Here we report for the first time, the Na-flux GaN growth with the necking technique by newly developed "GaN point seed", and we present typical structural characteristics of the grown crystals, which indeed prove the extremely high quality.

The GaN point seed was established by mounting a sapphire plate ($430 \mu\text{m}$ in thickness) with a small hole ($0.5 \sim 1 \text{ mm}$ in diameter) freely on a GaN template ($10\text{-}\mu\text{m}$ -thick (0001)GaN film on (0001) sapphire substrate). The GaN point seed was placed in an alumina (Al_2O_3) crucible, and the starting materials of metallic Ga, metallic Na, graphite grains, and Sr were added to the crucible in an Ar-filled glove box. Growth temperature and pressure were fixed at $890 \text{ }^\circ\text{C}$ and 4.0 MPa , respectively. Growth period were varied from 200 to 600 h. The crystallinity and dislocation density of grown GaN crystals were evaluated from X-ray rocking curves and panchromatic cathodoluminescence (CL) measurements.

Prism-shape and well-faceted bulk GaN single crystals without cracks could be obtained and its size reached as large as 0.85 cm in width and 1 cm in length, i.e. growth rates were $28 \mu\text{m/h}$ for both side of $\langle 11\text{-}20 \rangle$ direction and $17 \mu\text{m/h}$ for $\langle 0001 \rangle$ direction; this is the largest size reported by this method. The GaN single crystal grown with $0.03 \text{ mol}\%$ Sr addition has $c//$ and $c \perp$ full width at half maximum (FWHM) values as low as 42.8 and 32.5 arcsec , respectively; showing high crystallinity. After slicing the crystals parallel to a (0001) face and a chemical-mechanical polishing (CMP) process, CL measurements were performed to estimate the dislocation density in GaN samples. From panchromatic CL images, no dark spots deriving from dislocations could be seen in $120 \mu\text{m} \times 120 \mu\text{m}$ area. Furthermore, same measurements were performed at more than ten points across the wafer, and same results were obtained, indicating that large area of the wafer is free from dislocations.

Detail CL measurements near the interface between the seed and the grown crystal suggested that almost all dislocations were bent as soon as growth started and terminated at the sapphire sidewall in the hole. This elimination process of dislocations is quite similar to the necking technique in the Czochralski (CZ) growth of a dislocation-free Si ingot. We concluded that the necking technique in the Na flux method allows the elimination of dislocations propagating from a seed layer and could become a key breakthrough for fabricating large GaN crystals free from dislocations.

Crystal Growth of AlN Single Crystal by Sublimation method

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Aluminum Nitride (AlN) is a promising substrate material for Al-rich III-Nitride devices for use as deep ultraviolet light emitting diodes (DUV-LED) due to its superior transparency performance in the Deep UV region, high thermal conductivity and small mismatch with Al-rich III-nitride. Previous researchers [1,2] have already shown the sublimation-recondensation growth method to be a promising technique to grow large and high quality crystals.

In this study, we have also investigated the growth of AlN single crystal by the sublimation-recondensation growth method [3] and high transparency crystals from the visible to deep UV region have recently been produced. Our presentation will report our results so far with a comparison of the growth between the r-plane seed and c-plane seed.

The details of our growth experiments have been previously published by our former co-researchers [3]. The initial focus of our work was on the enlargement of the crystal size with AlN single crystals of over 40 mm in diameter having been achieved. However these initial crystals contained many impurities (mainly oxygen, silicon and carbon) which caused optical absorption as well as many defects as a result of the mismatch between AlN and SiC used as the seed.

Therefore we have recently focused on the improvement of the crystal quality through adjustment of the growth conditions and improvement of the crystal growth equipment. As a result we have grown crystals with excellent transparency in the UV region. Our results also show some interesting information about the color of the crystal grown on the r-plane seed having a clear yellowish color, while the crystal grown on the c-plane seed has a high transparency (absorption coefficient: $<14\text{cm}^{-1}$) from the deep UV to visible region.

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HVPE grown GaN: conducting and insulating substrates of various polarities

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III-nitride devices such as UV-blue-green emitters, high-power transistors and solar blind detectors have become commercial realities relying almost completely on heteroepitaxial approaches by employing a variety of substrates including, sapphire, silicon carbide and silicon. However, their performance is significantly limited by the structural quality of these materials as a result of the well-known disadvantages of the heteroepitaxy. In order to mitigate these challenges, several bulk GaN growth techniques have emerged and demonstrated significant progress during last few years. A brief comparative summary of the most promising ones will be presented but what could be summarized is that HVPE technique dominates today's bulk GaN market by providing substrates with diameter up to 3-inch and showing capacity to grow boules of about 10 mm.

Several HVPE manufacturing approaches for GaN substrates have been commercially implemented. Each of them employs different buffer layers or nucleation schemas, as well different growth recipes, and results in as-grown substrates with different thickness limitations. The doping alternatives, using either silicon or oxygen for achieving n-type conductivity and iron for achieving resistivity in wide ranges, respectively, were found to alter significantly the electrical properties of the substrates, while the optical quality was maintained until reaching the degeneration status. The defect ensembles and the charge transfer mechanisms were systematically evaluated in order to understand the Fe compensation mechanism and to achieve semi-insulating material with controllable characteristics. In addition, the doping variation has shown an unexpected strong influence on epitaxial optimum growth window likely via affecting the lattice parameter of the substrates.

Recent advances in the research and development of a variety of optoelectronic and electronic devices produced on HVPE bulk nitride materials with different conductivities have resulted in a significant improvement of device performance for a number of applications. This motivates the increased demand for HVPE nitrides, which should boost the material availability and will drive down the production cost. In this talk, a give brief snapshots of recent device achievements by using GaN bulk materials will be given. The focus will be on the substrate material properties with regard to the epitaxial requirements, such as surface orientation, polarity, miscut and doping.

GaN Quantum Devices for Infrared Optoelectronics

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Intersubband (ISB) devices rely on electronic transitions between quantum confined states, so that the desired wavelength of operation can be tuned via band engineering of semiconductor nanostructures. The capabilities of the ISB technology has been demonstrated in the mid-infrared and far-infrared spectral region using As-based materials like GaAs/AlGaAs and InGaAs/AlInAs-InP. The extension of ISB optoelectronics towards the near-infrared range is interesting for the development of ultrafast photonic devices for optical telecommunication networks, as well as for the implementation of a variety of chemical and biological sensors. The GaN/Al(Ga)N system is an excellent candidate for this application thanks to the large conduction band offset (about 1.8 eV for GaN/AlN) and sub-picosecond ISB relaxation time. Furthermore, the mechanical/chemical robustness and visible transparency open new possibilities in the domain of sensorics. This presentation will summarize the latest achievements in terms of growth and characterization of GaN/Al(Ga)N ISB devices (quantum cascade detectors, switchers, light emitters) operating in the 1.3-5 μm wavelength range. In particular, we will address the growth by plasma-assisted molecular-beam epitaxy, the challenges associated to strain relaxation and doping in nanostructures, and their effect on the structural properties, band engineering and electrical performance.

On the other hand, there is an interest to push the III-nitride ISB technology to longer wavelengths, in particular to the THz frequency range. The relevance of this spectral region for applications like security screening, quality control or medical diagnostics has driven extensive efforts to develop optoelectronics components. In this field, the large GaN LO-phonon energy (about three times that one of GaAs) opens prospects for ISB devices operating at room temperature and covering the infrared band that is inaccessible to As-based semiconductors due to Reststrahlen absorption. We have experimentally demonstrated that the mid-infrared domain up to 10 μm wavelength can be covered by GaN/AlGaIn quantum wells, and asymmetric-quantum-well superlattices make it possible to reach the far infrared. However, the spectral extension towards these long wavelengths requires a reduction of the polarization-induced internal electric field intrinsic to nitride heterostructures, which sets new material and design challenges.

Intersubband transitions at atmospheric window in $\text{Al}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$ multiple quantum wells grown on GaN/sapphire templates adopting AlN/GaN superlattices interlayer

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Recently, an increasing research effort has been directed towards the investigation of intersubband transitions (ISBTs) in $\text{Al}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$ multiple quantum wells (MQWs). Quantum well infrared photodetectors, quantum cascade lasers, quantum cascade detectors, and electro-optic modulators have been demonstrated using ISBTs in $\text{Al}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$ MQWs. However, most researches on GaN-based ISBTs are reported on the optical communication wavelength of 1.3 and 1.55 μm . Few efforts have been made on the atmospheric window of 3-5 μm , which is an important mid-infrared region for sensing and detection.

In this study, defects and strain control in $\text{Al}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$ MQWs for ISBTs at atmospheric window grown on GaN/sapphire templates by metal-organic chemical vapor deposition have been investigated adopting strain modulation technique with AlN/GaN superlattices (SLs) interlayer. It is found that cracking in the MQWs can be effectively avoided adopting AlN/GaN SLs interlayer. It is demonstrated that AlN/GaN SLs interlayer acts as a flexible layer and relieves most of the tensile strain through buried micro-cracks in AlN/GaN SLs interlayer. The intersubband absorptions at 3.6~4.1 μm wavelength region have been observed on the crack-free $\text{Al}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$ MQWs. Our results open up prospects to realize crack-free and high quality $\text{Al}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$ MQWs on GaN/sapphire templates for ISBTs devices at 3-5 μm atmospheric window.

Growth by ammonia source Molecular Beam Epitaxy of an AlGa_{0.3}N/GaN HEMT with an InGa_{0.1}N back-barrier

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Gallium Nitride is a very promising candidate for high power high frequency applications, especially in High Electron Mobility Transistors (HEMT). In this kind of devices the key point for high frequency performance is the confinement of the 2 Dimensional Electron Gas (2DEG). In order to improve the confinement it is possible either to grow the HEMT heterostructure on a thick AlGa_{0.3}N barrier or to insert a thin InGa_{0.1}N electrostatic barrier a few nanometers beneath the GaN channel. The main drawbacks of a thick AlGa_{0.3}N barrier are that it generally results in a lower crystal quality and it increases the tensile mechanical stress in the structure. On the other hand, InGa_{0.1}N back-barrier presents the advantage of being grown directly on the GaN buffer layer. The idea is to use the InGa_{0.1}N quantum well polarization field to raise the GaN conduction band level and so to improve the confinement of the 2DEG. The concept of InGa_{0.1}N back-barrier has been demonstrated using MOCVD growth technique [1], [2]. The aim of this study is to investigate another technique which has produced good quality HEMTs on various substrates like sapphire, silicon and silicon carbide, namely, the MBE growth with an ammonia source [3].

The AlGa_{0.3}N/GaN HEMT with an InGa_{0.1}N back-barrier has been grown in a RIBER 32 reactor. The structure consists in a 1μm thick GaN buffer layer on a Fe doped GaN on sapphire template, followed by a 2nm thick In_{0.1}Ga_{0.9}N back-barrier and the active layers: 10nm GaN channel, 1nm AlN spacer layer, 25nm Al_{0.3}Ga_{0.7}N barrier and 3nm GaN cap layer. Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM) confirm that the insertion of InGa_{0.1}N has no effect on the surface morphology. The combination of X-Ray Diffraction (XRD) scans and Transmission Electron Microscopy (TEM) images validate the good crystal quality of the heterostructure and the pseudomorphic growth of InGa_{0.1}N. This is also corroborated by the mobility measured by Hall Effect which is around 2100cm²/Vs for 2DEG carrier concentrations in the range of 8x10¹²cm⁻².

From a device point of view, by realizing transistors with a gate length of 3μm and a spacing source-drain about 13μm, the InGa_{0.1}N back-barrier does not disturb the DC transistor operation. To our knowledge, this is the first demonstration of an AlGa_{0.3}N/GaN HEMT with an InGa_{0.1}N back-barrier grown by ammonia source MBE.

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Regrown Source/Drain for Enhancement-Mode AlN/GaN MOSHFETs on Si substrate Exhibiting High Current and Transconductance

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AlGa_{0.3}N/GaN high electron mobility transistors (HEMTs) on silicon (Si) have potential applications in high-frequency and high-power electronics. Excellent device performance has been demonstrated for depletion-mode (D-mode) HEMT on silicon substrates. Enhancement-mode (E-mode) GaN HEMTs are desirable for many circuit applications. Specifically, normally off operation is preferred in power switching for safety and reduced power consumption. Combination of D-mode and E-mode HEMTs offers the simplest circuit configuration. Recently, high-performance normally-off AlN/GaN HFETs on Si substrates have been demonstrated. However, the high potential barrier of AlN makes it difficult to form a low contact resistance to the channel. Selectively regrowth of source/drain technology is one of the techniques to mitigate this problem, which has been used in both strained Si PMOSFET and AlN/GaN HEMT on SiC.

In this paper, normally-off AlN/GaN metal-oxide-semiconductor hetero-structure field-effect-transistors (MOSHFET) are reported. The AlN (1.5 nm)/GaN heterostructures were grown in an AIXTRON 2000 MOCVD system on 2-inch high resistivity Si (111) substrates. The epitaxial structure consisted of, a 40-nm AlN nucleation layer, 8 periods of AlN (11 nm)/Mg:Ga_{0.3}N (23 nm) super-lattice buffer, 2 cycles of GaN layer (600 nm)/low temperature AlN (20 nm) buffer, a 125-nm Mg doped GaN layer, 875-nm GaN buffer layer, and a 1.5-nm AlN barrier layer which was capped with 1-nm GaN. After the initial growth, the epi-wafer was patterned with an 84-nm-thick SiO₂ layer as a regrowth mask for the source/drain. The source and drain regions were etched to a depth of 120 nm from the sample surface by Cl₂-based ICP dry-etching. Then the patterned sample was reloaded into the MOCVD chamber for selective regrowth of a n⁺-Ga_{0.3}N ohmic contact layer. The growth temperature and Si doping concentration must be optimized as they have great influence on the surface morphology of the regrown regions. Optimum growth temperature and Si doping concentration were found to be 1090°C and 6×10¹⁹/cm³, respectively.

After regrowth of the n⁺-Ga_{0.3}N drain/source, the SiO₂ mask was removed. A 6-nm Al₂O₃ was deposited at 300°C in an Atomic-layer-deposited (ALD) chamber, then Ni/Au were deposited as gate metal. On top of gate metal, another 20-nm Al₂O₃ was deposited as passivation layer. Then the sample was annealed at 400°C for 10 minutes to enable gate sinking. The Al₂O₃ in the ohmic contact region was removed by HF solution. Finally, a Ti/Al/Ni/Au multilayer stack was deposited as source and drain electrodes. No thermal annealing was needed for the formation of ohmic contacts. From transmission line method (TLM) measurements, the metal/n⁺-Ga_{0.3}N contact resistance was 0.15 Ω-mm.

I_{ds}-V_{ds} characteristic of a MOSHFET device demonstrated a record-high current of 860 mA/mm at V_{gs}= 2 V. This device also exhibits a peak G_m of 509 mS/mm at V_{ds}= 4 V. To our knowledge, these DC values of MOSHFET are among the best demonstrated E-mode GaN MOSHFETs/HFETs on Si substrates.

Growth Optimization of AlGa_N/AlN/GaN HEMT Epitaxial Structures by MOCVD

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The goal of this work was to identify the growth conditions for AlGa_N/AlN/GaN epitaxial HEMT structures with high electrical characteristics of the 2-dimensional electron gas (2DEG).

The structures were grown by LP MOCVD on sapphire and SiC substrates. Hall-effect measurements were performed on the structures at room temperature to investigate 2DEG density and mobility. Structural properties were characterized by X-ray diffraction measurements.

When growth on sapphire, a 30 nm-thick (Al)Ga_N nucleation layer was deposited at 540°C on the substrate. Growth on SiC substrates began with deposition of a 50 nm-thick HT-AlN layer. The subsequent stages were practically the same in both processes – growth of 2 μm HT Ga_N layer, interfacial AlN layer (0,7 nm), AlGa_N layer-180 nm (Al~30%).

It was found that in all cases the 2DEG electron mobility depends mainly on the growth conditions of AlN interfacial layer and upper AlGa_N layer. High electron mobility in 2DEG can be reached even if the structural perfection of underlying Ga_N layer is not so high (even relatively high value of XRD-rocking curve full-width-at-half-maximum (FWHM) for the Ga_N layer as 320 arcsec for the (002) peak is tolerable).

This fact enables to grow high-resistance Ga_N layer and layer-substrate interface without any intentional compensation with deep-level impurities but only by some change of structural perfection of Ga_N layer (within the range of FWHM from 240 to 320 arcsec) that was effected by variation of Al content in AlGa_N nucleation layer.

Meanwhile, the best 2DEG electrical characteristics were obtained when AlN interfacial layer and upper AlGa_N layer were grown at relatively low temperature (1000°C) and V/III ratio (~1000) with growth rate no more than 7 nm/min

As a result of this optimization 2DEG electron mobility more than 1800 cm²/V.s at electron density of 1.4x10¹³ cm⁻² were obtained for HEMT structures with 30% Al content that gives sufficiently low sheet resistance of less than 250 Ω/sq. Such improvement in the electrical properties of HEMT structures with relatively low Al content is very attractive for device performances.

Kinetics of metal-rich PA MBE of AlGaN-heterostructures for UV-optoelectronics

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Development of ultra-violet (UV) optoelectronics within the challenging UV-B and UV-C wavelength ranges ($\lambda < 300$ nm) is one of the most promising areas of application of plasma-assisted MBE (PA MBE) due to its possibility of accurate controlling the thickness, composition and morphology of AlGaN quantum-well (QW) heterostructures on the atomic scale over the entire composition range. Recently, PA MBE grown optically-pumped AlGaN laser structures emitting at $\lambda \sim 300$ nm with the lowest threshold power density ($P_{th} \sim 800$ kW/cm²) for the structures grown directly on c-sapphire have been reported [1] as well as the 295 nm UV LEDs of 0.35 mW in cw output power [2]. Currently, the better results on photopumped UV lasers ($\lambda = 267$ nm, $P_{th} = 126$ kW/cm²) have been obtained only on the MOVPE structures grown on bulk AlN substrates [3].

This paper reports on elaboration of PA MBE of Al_xGa_{1-x}N-based QW-structures with high Al-content (up to 50% in the QW) grown directly on c-sapphire. Different elements of the structure design are considered consequently in detail along with the advanced growth approaches developed for each element. Special attention will be paid to the growth conditions of (i) AlN nucleation layers with suppressed generation of threading dislocations (TD), (ii) (2-3)- μ m-thick AlN buffer layers with atomically smooth droplet-free morphology (rms=0.46 nm) grown under the extremely metal-rich conditions ($F_{Al}/F_{N^*} = 1.4-1.5$), using periodic interruptions of Al-flux controlled precisely by the laser reflectometry, (iii) cladding and waveguide AlGaN layers also possessing the atomically-smooth droplet free morphology which was ensured by accurately established phase diagram of metal(Ga)-rich conditions within the temperature range $T_S = 680-750^\circ\text{C}$. Employing 3-nm-thick strained GaN insertions and AlGaN/AlN superlattices in the Al(Ga)N buffer layers along with control of their growth conditions led to the significant decrease of TD density down to 10^8-10^9 cm⁻² in the top MQW region.

An asymmetric position of the optically active AlGaN QWs, fabricated by a sub-monolayer digital alloying technique (SDA) with the accuracy better than 1 mol.%, was chosen in the waveguide of laser heterostructures to increase optical confinement factor. In addition, we demonstrate the pronounced increase of integral photoluminescence (PL) intensity in the samples with the MQW active region grown at some deviation from the ideal 2D morphology to 3D one, which is controlled by the F_{III}/F_{N^*} ratio. The nitrogen-rich growth conditions stimulate appearance of compositional inhomogeneities which provide efficient localization of the photoexcited carriers and suppress their transport to non-radiative centers at TD's. However, the optimum stoichiometric conditions have been found for photopumped lasers to ensure strong enough PL intensity and narrow PL emission band necessary for lasing.

Finally, the AlGaN-based MQW structures grown on c-sapphire using the above approaches are presented, which demonstrate optically-pumped lasing within the wavelength range $\lambda = 270-303$ nm with the minimum $P_{th} \sim 590$ kW/cm² ($\lambda = 289$ nm).

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MBE growth and applications of cubic AlN/GaN quantum wells

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Group III-nitride-based optoelectronic and electronic devices, which are commercially available at the market, are grown along the polar *c* direction, which suffer from the existence of strong “built-in” piezoelectric and spontaneous polarization. This inherent polarization limits the performance of optoelectronic devices containing quantum well or quantum dot active regions. To get rid of this problem much attention has been focused on the growth of non- or semi-polar (Al,Ga,In)N. An alternative way to eliminate polarization effects is the growth of cubic (100) oriented III-nitride layers. However, since cubic GaN is metastable and no cubic GaN bulk material exists in nature, heteroepitaxy with all its drawbacks due to lattice mismatch is necessary to grow this material. Due to the low lattice mismatch to cubic GaN the substrate of choice for the growth of cubic III-nitrides is 3C-SiC (001).

In this contribution the latest achievements in the molecular beam epitaxy of phase-pure cubic GaN, AlN and their alloys grown on 3C-SiC substrates is reviewed [1]. The structural and optical properties of cubic nitrides, cubic Al(Ga)N/GaN heterostructures, cubic GaN/Al(Ga)N multiple quantum wells (MQW) and superlattices (SL) will be shown [2], and the absence of polarization fields in cubic nitrides will be demonstrated [3]. Intersubband absorption and photoluminescence of cubic GaN/ Al(Ga)N quantum wells is studied experimentally and theoretically over a wide spectral range. By changing the quantum well thickness it is possible to tune the intersubband absorption peak wavelength from 1.4 μm (214 THz) to 63 μm (4.76 THz). Comparing the experimental results with simulations based on the effective-mass model the GaN/AlN conduction-band offset is demonstrated to be higher than 1.2 eV [4]. The best fit with the experimental data is achieved for a conduction-band offset of 1.4 eV and for a GaN effective mass of $0.11m_0$.

Resonant tunnelling diodes (RTD) of cubic AlN/GaN show a pronounced negative differential resistance at about 1.2V with a peak-to-valley ratio (PVR) of 1.3 to 2.7 at room temperature [5]. The experimental data are in good agreement with calculated I–V curves showing only a small deviation of 0.3V of the resonance peak voltage. We find a decrease of the PVR when the I–V characteristic is measured repeatedly with short time intervals between the voltage-cycles. However, the I–V characteristics can be recovered fully when the diodes are illuminated by UV-light indicating charge trapping in our devices.

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GaN/(Al,Ga)N heterostructures grown on (Zn,Mg)O by ammonia source molecular beam epitaxy

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Gallium nitride (GaN) based light emitting diodes (LEDs) have reached outstanding performances in the near UV-blue range. However, the quality of GaN epitaxial layers is still limited by high density of threading dislocations. This is mainly induced by the large lattice mismatch between GaN and sapphire, which is currently the most widely used substrate for GaN growth.

Zinc Oxide (ZnO) appears to be an alternative choice [1] as a substrate because it has the same crystallographic structure with GaN along with close lattice parameters and similar thermal expansion coefficients. For instance, an In_xGa_{1-x}N layer with an indium concentration of x~17% can be lattice-matched with a (0001)-oriented ZnO substrate. This could lead to the realization of InGaN templates that are more adapted to the growth of InGaN/GaN based LEDs with high In content (i.e. emitting above 500-nm). Moreover, high quality non polar ZnO substrates are also commercially available, which could be another alternative approach for the fabrication of GaN-based LEDs avoiding polarization discontinuities-related problems. However, strong interfacial reactions and atomic diffusions at high temperatures become one major problem when growing nitrides on ZnO. Our objective is then to develop a low temperature growth process for the realization of GaN/ZnO heterostructures by using molecular beam epitaxy (MBE).

In this study, we have grown GaN layers along both nonpolar (a[11 $\bar{2}$ 0]) and polar (c[0001]) orientations. In the nonpolar case, we have grown GaN on Zn_xMg_{1-x}O templates which consist of ~200nm Zn_{0.74}Mg_{0.26}O layers deposited on r-plane (1-102) sapphire. A series of nonpolar GaN/(Al,Ga)N multi-QWs (MQWs), with QW thickness range from 0.5 nm to 7 nm, were grown on top of 1 μm-thick GaN layers [2]. The a-plane orientation of the GaN layers has been confirmed by X-ray measurements. The optical properties were studied by low temperature photoluminescence (PL). As expected, no sign of quantum confined Stark effect (QCSE) is observed in the complete multi-quantum well (MQW) series. In addition, we demonstrate that the X₂ exciton (i.e. mainly polarized along[0001]) appears as the main contribution to the QW luminescence [2].

The growth of c-plane GaN layer has been carried out on commercially available bulk c-plane ZnO substrates. Before the growth, a flat ZnO surface with atomic steps was obtained after annealing. A two-step process was developed including a GaN buffer layer grown at ~ 600°C and a GaN layer grown at ~ 800°C on this buffer layer. We demonstrate that a surface roughness can be as low as 1nm while the GaN band-edge emission dominates the PL spectrum.

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Molecular Beam Epitaxy of High Electron Mobility InN and High Quality In_xGa_{1-x}N Layers (0≤x≤1)

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InN has the narrowest direct bandgap of 0.63 eV at room temperature and the smallest electron effective mass among III-nitrides. The ternary alloys such as InGaN and InAlN cover a very wide wavelength region and thus provide the possibility for fabricating a lot of optoelectronic devices such as high efficiency solar cells and laser diodes in optical communication wavelength regions. All of these devices require high quality materials, i.e. either InN or its alloys. However, the epitaxy of InN and its alloys is very difficult due to the low maximum epitaxial temperature (500-600°C) and the large lattice mismatch of InN with common buffer layers and/or substrates (for example 11% with GaN and 25% with sapphire). Much efforts are necessary to improve the quality of InN and InGaN in order to satisfy the requirement of devices. In this talk, we would like to report our recent progress on growth of high quality InN layer and InGaN layers with In composition varying from 0 to 100%.

At first, we have set up a method called boundary-temperature-controlled epitaxy, where the growth temperature of InN is controlled at its maximum one, to get high electron mobility InN layers. The Hall-effect measurement shows a recorded directly-probed mobility of 3010 cm²/Vs and a residual electron concentration of 1.77 cm⁻³ at room temperature, which corresponds to a mobility of 3280 cm²/Vs and a residual electron concentration of 1.47×10¹⁷ cm⁻³ in InN bulk layer. The enhanced electron mobility and the reduced residual electron concentration are mainly due to the reduction of threading dislocation density. The obtained Hall mobilities are in good agreement with the theoretical modelling by the ensemble Monte Carlo simulation.

Then, In_xGa_{1-x}N alloys (0≤x≤1) have been grown on GaN/sapphire templates. Growth temperature controlled epitaxy (GTCE) was proposed to modulate In composition. The GTCE method shows two advantages over the common growth method: (1) The growth can be kept at the highest temperature for each for In_xGa_{1-x}N alloy with specific *x*. This leads to better crystalline quality and surface due to enhanced migration of adatoms, in particular for samples with relatively large In composition. (2) The growth is always kept under In-rich condition. This is helpful to obtain smooth surface since the strong enhancement of adatom diffusion length can be realized underneath a thin In layer on the growing surface. As a result, In_xGa_{1-x}N alloys (0≤x≤1) with good crystalline quality, surface morphology and optical property have been achieved. Moreover, The bandgap energies of the In_xGa_{1-x}N alloys have been evaluated by optical transmission spectroscopy, where effect of residual strain and electron concentration on bandgap energy shift has been considered. Finally, a bowing parameter of ~1.9±0.1 eV has been obtained by the well fitting for the In-composition dependent bandgap energy.

In-situ monitoring of InGaN growth using DERI method

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Recently, a new RF-MBE growth method called DERI (Droplet Elimination by Radical beam Irradiation) was proposed for the growth of InN [1]. This growth method is composed of two series of growth steps: first, an In-rich growth step (MRGP: Metal-Rich Growth Process), followed by a nitrogen radical (N*) beam irradiation step (DEP: Droplet Elimination Process). Typically, excess In during the MRGP step forms droplets on the surface. These droplets are eliminated by their transformation to epitaxial InN during the DEP step. The growth process can be accurately characterized by *in-situ* monitoring techniques using RHEED. Therefore, the DERI method enables the simple and reproducible growth of high-quality InN. We also demonstrated the application of DERI method to the growth of InGaN alloys [2]. In the MRGP, InGaN was grown under a metal rich (Ga+In>N*) condition by supplying Ga, In and N* simultaneously. Here, the preferential capturing process of Ga from Ga/In wetting layers was occurred, and then In atoms swept out from the growing interface stayed either as wetting layer or as droplets on the surface. This swept In on the InGaN underlayer was transformed to InGaN by the following DEP step with only N* and Ga supply. In this paper, we study the *in-situ* monitoring of the InGaN growth during the DERI process.

During the MRGP step, only In was supplied, and then InGaN was grown by the subsequent N* and Ga supply during the DEP step. Here, the composition of InGaN was determined by the flux ratio of Ga/N* in DEP. The InGaN growth process also can be monitored *in-situ* using the variation in intensity of the RHEED diffraction pattern. We define the time of In supply in MRGP as T and the time required to recover the RHEED intensity under nitrogen radical beam irradiation in DEP as τ . Under a constant T of 30 s and plasma power of 200 W, τ was measured as a function of growth temperature for InGaN with various In composition. It is found that with the increase of the growth temperature, τ decreased for Ga-rich InGaN and increased for In-rich InGaN respectively. These results suggest that the proposed monitoring technique during InGaN growth by DERI can be useful for the analysis of thermal desorption process of In and Ga atoms from InGaN.

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True green LDs on c-plane: growth and LD parameters

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Direct green emitting laser diodes (LDs) with an output power over 50mW, wavelengths larger than 515nm and reasonable lifetimes are highly desired by the market for embedded laser projection.

There are several epitaxial challenges on the way to green emitting InGaN LDs grown on c-plane GaN departing from the already commercially available blue emitting InGaN LDs.

The main challenge is that the realization of green emitting quantum wells (QWs) with metal organic vapor phase epitaxy (MOVPE) enforces growth in an unfavorable growth regime. Low growth temperatures have to be applied for sufficient indium incorporation which in addition gives the inconvenience of very low decomposition efficiency for ammonia. Green emitting QWs with indium contents > 30% can be successfully grown on freestanding GaN c-plane substrates in spite of experiencing a lattice mismatch larger than 3%. Both, the low growth temperature and the high compressive strain of green QWs, lead to the undesirable appearance of crystal defects and inhomogeneous indium incorporation. Therefore it is necessary to investigate the mechanisms of defect formation and inhomogeneous indium incorporation in detail to improve the crystal quality and therefore the optical properties such as gain.

In this contribution, we will present measurements of specially designed test structures that enable TEM and XRD analysis of defect type and relaxation processes appearing in green emitting QW layers.

Furthermore, different origins of inhomogeneous indium incorporation in indium rich InGaN QWs will be discussed. We will show results on artificially created mesa structures that allow realization of controlled local strain relaxation to cause inhomogeneous indium incorporation.

Finally, we will present recent laser data of green emitting LDs with emission wavelength above 515nm, 50mW output power, a WPE over 5% and lifetimes up to 10.000 hours.

Development of semipolar GaN-based laser diodes

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The availability of free-standing substrates, sliced from bulk GaN crystals in various planes, opened the opportunity to use epitaxial growth and material characteristics different from those for conventional c-plane structures. In particular, green laser diodes (LDs) with output power >50 mW grown on semipolar substrates were achieved. Overall, the growth of semipolar InGaN layers used for QWs and waveguides, has wider process window than in c-plane for incorporating indium with sufficient concentration. In terms of material properties, we demonstrated that semipolar-plane InGaN quantum well (QW) can provide ~2x higher optical gain thanks to small quantum confinement Stark effect along with narrower linewidth and polarization anisotropy of light emission. We have also observed the threshold characteristic temperature T_0 to be much higher than that for c-plane green LDs – an important advantage for mobile device applications, where heat-sink capability is limited. In addition, we have found that the carrier injection into several deep-blue and green QWs is much easier to achieve in semipolar-plane diode thanks to faster ballistic hole transport allowed by lower built-in electric fields.

The misfit dislocation (MD) formation is more likely in semipolar strained heterostructures. While the threading dislocations (TDs) are not critical for a light emitter performance, the MDs in LDs are not only sources of nonradiative recombination but also during the growth they cause surface and interface hatch-like roughening, that in turn causes light decoupling from the waveguide and increases QW composition nonuniformity negatively impacting optical net gain. The formation of MDs is assisted by glide of TDs, therefore one way to avoid MDs is using substrates with low enough TD density. Another way to address the MD formation is to keep them far enough from the active region. For this we grow relaxed InGaN waveguide and an MQW active region on top of it, followed by the second InGaN waveguide. This stack itself is grown coherently while dislocations remain only in the interfaces between waveguides and cladding layers. One more solution is inserting strain compensating layers with tensile strain between QWs and in InGaN waveguiding layers. Growth of tensile Al(In)GaN barriers between MQWs prevents active region relaxation. In the waveguide core, adding Al-containing layers is expected to reduce not only strain, but also average refractive index. However, the InGaN refractive index increases superlinearly with In concentration, therefore when In concentration in InGaN layers is sufficiently high, one can achieve strain compensation without degradation of optical confinement. This approach is more attractive for LDs with deep blue-green light emission.

Optical loss is another important issue in III-N LDs, in particular green LDs where optical gain is smaller. We have determined that the main source of internal optical loss is light absorption in acceptor-bound holes, being in semipolar LDs as high as in c-plane ones. In order to achieve sufficient p-conductivity we need a high concentration of Mg, while most of the holes remain Mg-bound due to high activation energy. The optical loss may be reduced via using an asymmetric waveguide design that shifts the optical mode away from p-side. Also, the part of the waveguide close to the optical mode peak can be lightly doped, with the cost of some increase in operation voltage.

GaN substrates grown by HNPS method in Multi-Feed-Seed configuration and their application for laser diode arrays with variable wavelength

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It is shown that the High Nitrogen Pressure Solution (HNPS) method in Multi Feed Seed (MFS) configuration based on direct conversion of large seeds grown by HVPE into high pressure material allows obtaining GaN crystals with low dislocation density, flat crystallographic planes and very high and uniform electrical conductivity. Such crystals are optimum substrates for laser diodes and laser diode arrays also due to their lowered refractive index preventing optical modes to penetrate into the substrate.

In this work the dependence of indium content in MOVPE grown InGaN on the substrate miscut angle was used for fabrication of laser diodes emitting light of different wavelengths integrated in a single substrate grown by HNPS method. It is shown that the PL wavelength decreases with increasing vicinal angle and it is related to the In content variation. Therefore the substrates were patterned by photolithography and ion etching to achieve surface profile with differently angled regions.

The patterned free standing GaN substrates with separate regions angled by 0.35 to 0.85° to the c-plane were used to fabricate ridge waveguide laser diodes. Each laser stripe was placed inside one of the angled areas. Lasing wavelengths of 405 and 401 nm were obtained for devices grown in these regions.

Structure and stability of InGaN-based active regions in laser diodes grown by MOVPE

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Structural quality of MQW-based active regions has a significant impact on the performance of (Al,In,Ga)N-based light emitting devices. While the influence of QW properties and growth conditions on the output power and the threshold current density is subject of many publications, our study deals with the effect of quantum barrier growth on the structural properties of InGaN-based active regions as well as the characteristics of optically pumpable laser structures. For LEDs emitting in the blue and green wavelength region it has been shown, that deposition of GaN quantum barriers at slightly increased temperatures improves the interface smoothness of the active region and leads to a reduced v-pit density. In addition, an improvement of the structural as well as electrical properties of devices can be achieved by a replacement of GaN barriers in the active region by InGaN. In our experiment we combine both findings – a replacement of GaN barriers by InGaN and a barrier growth at higher temperature compared to the QW. For this purpose active regions consisting of InGaN/InGaN DQWs emitting between 400 and 440 nm were grown on GaN substrates. In particular, two structures were compared: InGaN barriers and QWs deposited at the same temperature of about 800°C (LT-barriers) and InGaN barriers grown at 870°C (HT-barriers) with the QW deposition at 800°C. To reveal the influence of barrier growth temperature onto the structural and luminescence properties of the active regions the samples were analysed by high-resolution X-ray diffraction, transmission electron microscopy (TEM), cathodoluminescence (CL) and photoluminescence (PL).

It has been observed that HT-deposition of InGaN barriers leads to a significant thickness non-uniformity of the QWs as well as the corresponding barriers, whereas LT-barrier deposition results in a more uniform QW thickness. The high thickness fluctuations can lead to a stronger carrier localization in the HT-grown layers, which has been confirmed by temperature dependent PL measurements.

The heterostructures containing active regions were further processed to optically pumpable lasers. The devices containing HT-barriers show a significantly lower peak modal gain as a consequence of the higher lateral band gap non-uniformity. An increased optical threshold power density of about 600 kW/cm² was measured for these structures in comparison to 200 kW/cm² observed for lasers with LT-barriers. This result suggests that a uniform QW thickness results in a superior laser performance.

In our second experiment we analyzed the stability of the InGaN active regions during the subsequent deposition of p-type AlGaIn layers as used for the fabrication of current injection laser structures. Deposition of a p-type AlGaIn at 950°C has no impact on the structure of the active region with HT-barriers indicating its good stability. In contrast, CL analysis of samples with LT-barriers shows large areas with no CL intensity. TEM analysis reveals a complete QW decomposition in these regions with a formation of In-rich grains separated by voids. This QW decomposition can be avoided by a reduction of the p-AlGaIn deposition temperature by 30 K.

InGaN prepared by pulsed sputtering and its application to solar cells

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Recently, solar cells based on group III nitrides have attracted much attention because InGaN has high light absorption coefficients and energy band gaps that cover most of the solar spectrum (0.65-3.4 eV) [1,2]. However, the nitride solar cells suffer from the phase separation reactions during the growth of thick InGaN with high In concentrations which is necessary to make the film absorb a large amount of light. Recently, we have found that the use of a low temperature growth technique called pulsed sputtering deposition (PSD) allows us to suppress the phase separation reactions of InGaN and to obtain high quality epitaxial films [3,4]. In this presentation, we will discuss advantages in the use of the PSD low-temperature growth technique for the fabrication of InGaN-based solar cells.

Based on the data of device simulator, we chose n-GaN/InGaN/p-GaN double hetero-junction structures for solar cells [5]. Thickness of InGaN layers is approximately 200 nm. ITO and Ni layers were used as ohmic contacts for n-type and p-type GaN layers, respectively. The devices were characterized under AM1.5G light illumination at 100 mW/cm².

The RHEED images of InGaN layers showed clear streaky patterns for all the In compositions from 0% to 100%, which indicates that InGaN grows epitaxially with smooth surfaces. In the XRD 2 θ / ω curves for approximately 200-nm-thick InGaN films, 0002 diffraction is the sole peak and any other InGaN-related peaks were not observed. This fact indicates reduction in growth temperature lead to successful suppression of the phase separation reactions during the InGaN growth. Room-temperature photoluminescence spectra of the InGaN films exhibited intense near-band-edge emissions in the wavelength range from 1.7 eV to 3.4 eV, which covers whole visible spectrum region. To investigate the lattice strain in the InGaN films, X-ray reciprocal space mapping was performed. We found that InGaN layers with In contents less than 6% commensurately grow on GaN layers, while those with In contents over 17% showed clear lattice relaxation. This phenomenon is caused by the increase in the lattice mismatches between InGaN and GaN and is known to be alleviated by the use of the nearly lattice matched substrates such as ZnO [6]. Photovoltaic characteristics of the n-GaN/InGaN/p-GaN double heterostructures fabricated by PSD were investigated under AM1.5G illuminations. We found that the open circuit voltage (V_{oc}) decreases from 2.3 V to 0.9 V with increasing the In content of the InGaN layers upto 47%, which agrees well with the results of device simulations.

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III-N nanocolumn growth, properties and LEDs

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GaN nanocolumns were self-assembled by rf-plasma-assisted molecular-beam epitaxy (rf-MBE) [1], but self-assembling of the nanocolumns by rf-MBE introduces spatial variation in size and position of nanocolumn, resulting in the multi-color emission of the InGaN-based nanocolumn LEDs [2]. The nanocolumn diameter and position were controlled precisely by the selective area growth (SAG) technique for the GaN nanocolumns developed [3, 4].

In this study, GaN nanocolumns with the nanocolumn diameters from 62 nm to 853 nm were grown on GaN templates to investigate systematically the dislocation property of nanocolumn. The cross-sectional TEM images of the nanocolumns were observed, evincing that no new threading dislocations were generated at the boundaries between the nanocolumns and the underlying GaN template. Some nanocolumns were grown accidentally just on dislocation lines existed inside the GaN template; for wide nanocolumns, the dislocation reached to the top region of nanocolumn, but for narrow nanocolumns with the diameter, typically less than 300 nm, dislocations bent toward the side wall to be terminated and never propagated through the nanocolumn toward the upper region. Next the photoluminescence (PL) spectra of the red emission InGaN/GaN MQW nanocolumn arrays (600 to 660 nm in wavelength) were evaluated as a function of temperature from 4 K to 300K. For the 661nm emission nanocolumn, the PL integrated intensity ratio between 4 K and 300 K was evaluated to be 17.5%.

Then, uniform arrays in triangular-lattice of GaN nanocolumns were grown on GaN templates by the SAG of rf-MBE and at the top regions of nanocolumns, InGaN/GaN multiple quantum wells (MQWs) were integrated. InGaN-based LEDs with the nanocolumn array operated under the room temperature DC current injection, emitting the green light from 520 to 540nm. The emission-color of the nanocolumn arrays changed from blue to red with increasing the nanocolumn diameter from 137 nm to 270 nm at the same lattice constant of 400 nm [5]. Based on this finding, the monolithic integration of green and orange emissions nanocolumn LEDs was successfully demonstrated. In the experiment, the nanocolumn LED crystals with the nanocolumn diameters of 150, 190, 230 nm were grown on the same GaN template; the emission peak wavelengths changed from 541, 580, and to 597nm with the nanocolumn diameter. At the same time, In-rich In_{0.86}Ga_{0.14}N active layers were grown on the GaN nanocolumn arrays, followed by the growth of Mg-doped Ga-rich In_{0.3}Ga_{0.7}N cladding layers; periodic arrangement and dislocation property of the nanocolumns contributed to the high crystalline quality of the In-rich InGaN. Using the grown crystals, the nanocolumn LEDs were fabricated, achieving the successful operation of near-infrared (1.46 μm in wavelength) In-rich InGaN nanocolumn LEDs [6].

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Photodetection properties of single GaN nanowires

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Photodetectors based on nanowires (NW) picture new revolutionary technological applications for quantum electronics, optoelectronics, and sensor applications. In the case of GaN NWs the combination of their exceptional properties, namely the huge photocurrent gain, giant ultraviolet/visible contrast, practical bandwidth and environment insensitivity selectivity, renders GaN NWs particularly attractive for reliable and robust photodetector applications. However, a deeper understanding of the NW structural and electronic properties is a key issue for uncovering the role of surface processes in the light-matter interaction at the nano-scale.

In this work, we will present a broad study of the photocurrent phenomena in single defect-free GaN NWs, analyzing the effect of the contact nature (ohmic-ohmic, ohmic-Schottky or Schottky-Schottky), excitation power, light polarization, measuring frequency, and environment. Non-intentionally-doped and $n-i-n$ GaN NWs were grown by plasma-assisted MBE under nitrogen-rich conditions, and they were dispersed on a pre-patterned SiO₂/Si, in order to fabricate the single NW photodevices with electron beam lithography. The devices under study consist of defect-free single GaN NWs with a diameter of 30–40 nm and a length of 1 to 1.50 μm. In the dark, they display space-charge-limited current due to the surface-induced total depletion of the non-intentionally-doped sections. Devices with ohmic-ohmic contacts (symmetric $n-i-n$ structures with Ti/Al-based contacts) present current densities in the nA range, at least two orders of magnitude higher than in Schottky-Schottky devices (undoped structures with Ni/Au contacts). Under illumination, their photocurrent scales sublinearly with the excitation power, and their spectral response is relatively flat for excitation above the GaN bandgap and presents a visible rejection of more than six orders of magnitude.

The $n-i-n$ NW photodetectors are relatively insensitive to the measurement environment, either in vacuum or in the air, and present a relaxation time in the range of ms. In comparison, the non-intentionally-doped single NW devices with Ni/Au Schottky contacts display lower photocurrent gain with a remarkable sensitivity to the measurement environment: The gain decreases by about two orders of magnitude when devices are exposed to air, in comparison to measurements in vacuum. Furthermore, undoped devices present persistent photoconductivity in the range of minutes, with a slower response in the case of measurements in vacuum. These results indicate that the pinning of the Fermi level at the NW n -regions in $n-i-n$ devices grants certain insensitivity of the photodetector performance to the surface charge state. On the contrary, in non-intentionally-doped NWs, surface states play a major role on the photocurrent dynamics due to the unpinned Fermi level. In the air, adsorbed oxygen accelerates the surface processes, whereas in vacuum, due to the longer recombination time associated to the surface states, the NWs approach the behavior of two-dimensional GaN photodetectors.

Semi-polar GaN quantum wells grown on {11-2n} planes at the pyramidal tip of GaN nanocolumn arrays

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GaN nanostructures are of great interest for optoelectronic devices and in general as nanoelectronic building blocks. The capability of controlling the size, position, geometry and composition of individual nanoscale functional objects on a given substrate is one of the key requirements to realize future electronic and optoelectronic devices or sensors that are composed of such nano-objects.

In this study, we report on non-catalytic diffusion-assisted selective area growth (SAG) of GaN nanocolumns on Mo pre-patterned GaN(0001) and 6H-SiC(0001) substrates by molecular beam epitaxy (MBE). Position and size control is achieved by patterning arrays of apertures into a thin Mo layer by electron beam lithography. Using a wide range of diameters and pitches the influence of these parameters on the axial growth rate of the nanocolumns can be investigated and a contribution from the Ga diffusion on the Mo mask is put in evidence.

The tip of the nanocolumns is of pyramidal shape showing semi-polar {11-2n} ($n = 2.5-4$) facets. $\text{Al}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$ quantum wells (QWs) have been grown on the semi-polar {11-2n} planes. Multi quantum well (MQW) samples have been grown to get an insight into the growth rates along the semi-polar direction. The crystal quality of the nanocolumns and the semi-polar quantum wells is characterized by transmission electron microscopy (TEM). In addition, the optical properties of the semi-polar QWs are studied by photoluminescence spectroscopy.

The control of crystal polarity for MOCVD nanowire growth

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The crystal polarity is known to strongly impact the growth kinetics and morphologies of the epitaxial growth of polar wurtzite crystals. This phenomenon has been recently demonstrated in the case of homoepitaxial MOVPE growth of nitride nanostructures for which the substrate polarity imposes the shapes: *Ga-face polarity ([0001] direction) results in pyramidal-shaped nanostructures, whereas N-face polarity ([000-1] direction) results in catalyst-free hexagonal wires with a flat top c-plane surface* [1]. This result points out the importance of crystal polarity to control the growth of nitride nanostructures. Especially, we will show in this work the key-role of polarity to achieve the wire growth.

The growth of **N-polar GaN wires** has been reported on c-sapphire substrates [1,2]. In this case, a nitridation step is first performed on sapphire substrate to induce N-polar crystal growth. N-polar GaN wires present a hexagonal shape with flat top surface and exhibit a diameters around 400-800 nm (similar to wires grown in homoepitaxy on N-polar GaN substrates). Differently, we have recently performed the growth of **Ga-polar GaN wires** also on c-sapphire substrates. Such growth requires initiating Ga-polar growth by removing the surface nitridation treatment of the sapphire (similar wires can be grown on Ga-polar GaN templates). The Ga-polarity induces pencil-shape wires with sharp pyramids at their top. The wire growth with vertical m-plane facet formation is obtained combining low V/III ratio and highly reduced V-III precursor flows (two orders of magnitude lower than in standard growth) in order to favor the reaction-limited growth regime. These GaN wires present much smaller diameters (100-200 nm) and are free of extended defects as established by HRTEM and XRD measurements. Such wires exhibit excellent optical properties demonstrated by a narrow photoluminescence linewidth (1–3 meV at 5 K) of the near band edge positioned at the fully relaxed value (3.47 eV) relevant with the excellent crystalline quality of such Ga-polar GaN wires [3].

Finally, we have demonstrated the presence of **polarity mixing** inside single wires [1], that can play an important role for the growth of quantum wells (QWs). Such effect has been observed for the growth of InGaN/GaN QWs. The specific role of polarity on the structural and optical QWs features will be presented and will be related to the wire-based device characteristics [5].

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Scaling growth laws of self-induced GaN nanowires on Si substrates

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Semiconductor nanowires (NWs) are promising building blocks of future nanoelectronic, and nanophotonic devices. The self-induced NW formation is an attractive approach to ease their synthesis and improve functionality. In particular, the self-induced growth of GaN NWs on Si substrates has recently drawn much attention, since it provides a new way for monolithic integration of high quality GaN nanostructures with Si platform. The controlled synthesis of self-induced NWs requires, however, a deep understanding of their growth mechanisms, and the initial nucleation step in particular.

In the first part of the talk we present our experimental data on the MBE growth of GaN NWs on Si substrates, either covered with a crystalline AlN lattice mismatched layer [1] or with a SixNy amorphous interlayer [2]. In particular, we show that GaN never nucleates in the NW morphology. Rather, the nanostructures emerge as 3D nanoislands having a fixed shape, while the shape transformation to NWs occurs only after these islands exceed a certain critical size.

The second part is devoted to the growth modeling. We present a scaling model that explains the self-induced formation of GaN nanowires by the anisotropy of surface energies coupled with the scaling growth anisotropy. The model shows why GaN always nucleates in the form of nanoislands rather than nanowires. It also elucidates the physical origin of the island to wire shape transformation [3].

We also discuss a kinetic model showing why self-induced GaN nanowires synthesized by MBE obey the scaling growth laws. Our model explains the scaling behavior from kinetic considerations of the step flow radial growth and the shadow effect. Finally, we show how the growth models developed earlier for III-V and group IV “vapor-liquid-solid” NWs [4,5] can be applied to the case of self-induced GaN NWs. In particular, we present theoretical and experimental dependences of the GaN NW growth rate on the deposition time, Ga flux and surface temperature that demonstrate an excellent correlation with each other.

Overall, the scaling growth laws of self-induced GaN NWs should be considered fundamental; however, the scaling indices can be changed by the deposition conditions. This is paramount for better understanding of self-induced NW growth in general and provides a powerful tool to tune the GaN NW morphology to the desired properties.

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Localized surface plasmons enhancement of GaN/InGaN LED performance: problems and possible solutions

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It is known that deposition of Ag nanoparticles (NPs) on top of GaN/InGaN MQW structures can increase the IQE of luminescence by several times due to the coupling of MQW region with the electromagnetic field of localized surface plasmons (LSPs) associated with Ag NPs. The phenomenon could be of a vast practical importance for improving GaN LEDs performance. However, its application in real-life devices is hampered by several important problems. First, the spacial extent of efficient coupling does not exceed, according to theoretical estimates, more than about 40 nm, whereas, for efficient LED operation the thickness of the p-GaN emitter should be higher than about 100 nm. Secondly, the performance of structures with Ag NPs LSPs varies with time due to chemical interaction of Ag with the ambient atmosphere. Thirdly, the Ag NPs are conducting which creates problems when trying to use them with advanced LED designs, such as nanopillar structures. In this talk we will discuss possible approaches to solving these problems that have been tried in our laboratories. We show that, when using nanopillar structures with the pockets filled with Ag NPs, one can circumvent the main difficulty of placing the Ag NPs in close proximity of the MQW region. Two technologically viable approaches have been tried. In the first, the nanopillar structure has been prepared in the p-GaN cap by dry etching via Ni nanomask and the pockets of the structure filled with Ag NPs. In the second, the nanopillar structure was prepared in the n-GaN film underlying the MQW region and overgrown by MOCVD. In both cases enhancement of LED efficiency by several times has been demonstrated. The problem of stability of LSP enhancement could be solved by developing the technology of Ag/SiO₂ core/shell NPs prepared by sol-gel method. The diameter of the Ag cores and SiO₂ shells could be controllably varied in the 30-50 nm range and 2-20 nm range, respectively. This places the LSP resonance peak near 450 nm matching it to the blue MQW LEDs. We demonstrate that strong PL intensity enhancement can be achieved for MQW structures with Ag/SiO₂ NPs, describe the results of theoretical modeling and experimental studies of the efficiency of LSP coupling to MQW as a function of distance between the NPs layer and the QW for varying numbers of QWs. These studies seem to indicate that the extent of efficient spacial coupling covers the range of about 40 nm, in agreement with earlier calculations for Ag NPs. Measurements of the LSP-related PL enhancement stability over time due to Ag/SiO₂ core/shell NPs is better than for Ag NPs (no measurable changes in PL intensity over the period of several months as opposed to several weeks in the latter case). We also describe application of Ag/SiO₂ core/shell NPs to enhancement of PL efficiency of GaN/InGaN MQW nanopillar LED structures prepared by dry etching of planar diode structures using self-organized Ni nanomask.

Controlling (ferro)magnetism in III-Nitrides

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We summarize our recent work on controlling and elucidating the magnetism and exchange interactions in GaN and related systems grown by MOVPE and doped with either Fe [1-7] or Mn [8-10] and co-doped with donors (Si) [3,4,9] or acceptors (Mg) [3,7]. In particular, we show that a significant contribution of d orbitals to the bonding leads to the aggregation of Fe cations at the growth surface driving the systems to the state of condensed magnetic semiconductor (CMS), i.e. to a semiconducting matrix with Fe-rich nanoscale chemical or crystallographic phase separation [11,12]. The correlation between the presence of different Fe-rich phases with a peculiar and well-defined magnetic behaviour is highlighted together with ways for the realisation of a single-phase CMS. Furthermore, we demonstrate – by employing a range of nanocharacterisation tools - that Mn in GaN occupies random cation positions at least up to $x = 3\%$. We present experimental results on the determination of the coupling strength between Mn ions in GaN and show how by co-doping with Si the dominant interaction can be tuned from ferromagnetic to antiferromagnetic superexchange [9].

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Theory of transition metal aggregation and spin-spin interactions in III-nitrides

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Stimulated by recent progress in MOVPE and nanocharacterization of transition-metal (TM) doped GaN [1-6] we have employed *ab initio* (GGA + U) and tight binding (sp^3s^* and the Parmenter hamiltonian) approaches to elucidate the nature of TM incorporation, distribution, and magnetism depending on the growth conditions and co-doping by shallow impurities. Our recently published [4,7] and newer results [8] reveal that:

1. Owing to contribution of d orbitals to bonding, there is a strong attractive force between TM cations inside GaN. However, in contrast to previous expectations, we show the aggregation of TM cations is controlled by pairing energies at the growth surface rather than in the bulk of the films. By quantitative evaluation of these energies we elucidate why Fe-rich nanocrystals are formed [1-4], whereas Mn cations remain diluted [5,6] in GaN.
2. By studying the effect of Si and Mg co-doping in the bulk and at the growth surface we find out mechanisms by which the Fe aggregation is halted by co-doping with shallow impurities, as observed experimentally for (Ga,Fe)N:Si [1,2] and (Ga,Fe)N:Si [1,4]. We also provide a plausible explanation for the puzzling enhancement of the Fe aggregation when Mg is incorporated in a digital fashion [4].
3. We examine spin-dependent coupling between Mn cation pairs in GaN by *ab initio* and tight binding approaches. Our results explain a transition between ferromagnetic and antiferromagnetic superexchange on varying the Mn charge state as well as give direct information on the range of the magnetic coupling in dilute magnetic insulators.

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Scandium-based nitrides: new members of the III-nitride family

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Although great progress has been made on III-nitride devices over the last 20 years, significant challenges remain: the internal quantum efficiencies of green and UV-emitting devices are much lower than their blue-emitting counterparts, and these values drop at high current densities. These problems have motivated us to seek new nitride materials that can be used to extend device functionality. ScN is our first choice for use in novel III-nitride alloys, as its properties are close to those of conventional III-nitrides.

Usually, transition metals are not easily soluble in III-nitrides and tend to produce deep levels in the band gap. However, Sc is a significant exception to this rule. Our DFT calculations show that ScGaN alloys are stable across the entire composition range, retaining the wurtzite structure and a direct band gap at low Sc contents. However, the in-plane lattice parameter a increases significantly as the Sc content increases. This is accompanied by a ‘flattening’ of the crystal structure (decreased c/a ratio). Similar trends are predicted for ScAlN, except that Sc has only a moderate solubility in AlN. Both ScAlN and ScGaN offer prospects for use in UV light-emitting devices, while ScInGaN alloys can offer lattice-matched emission across the entire visible range.

There are several advantages to the growth of Sc-based nitrides. Unlike InGaN, ScGaN is stable at high temperatures and the substrate temperature does not strongly affect the incorporation of Sc. Sc also appears to catalyse the decomposition of N₂, as ScN growth rates are similar using either N₂ gas or a N₂ plasma source in molecular beam epitaxy (MBE). Additional variables are thus present compared to conventional III-nitride growth, including the possibility of varying the proportion of active nitrogen in the gas supply and choosing Sc-rich as well as Ga-rich or N-rich growth conditions. This offers greater freedom to achieve optimum growth conditions, which we find occur for Sc-rich growth. The greater in-plane lattice parameter also produces compressive stress, thereby preventing film cracking (a major problem for UV devices based on AlGaN).

We have recently demonstrated that high quality heteroepitaxial ScGaN and ScAlN films can be grown by plasma-MBE on GaN-(or AlN)-on-sapphire substrates. The use of a low dislocation density wurtzite-structure ‘template’ helps to stabilise the hexagonal crystal structure in ScAlN and promotes high quality film growth. High substrate temperatures (850 °C) are found to produce better quality ScGaN and ScAlN films and to promote increased lateral growth rates compared to lower substrate temperatures (600 °C). The dislocation densities in low Sc-content ScGaN films are limited only by those of the underlying GaN ‘template’ layer. For higher Sc contents, transmission electron microscopy studies show that basal-plane stacking faults and associated strain-relieving dislocation loops can form. At low Sc contents, intense luminescence can be observed from some ScGaN films, indicating its promise for optoelectronic applications. Here, we will present a full overview of the growth and applications of this exciting new class of III-nitrides.

Molecular beam epitaxy of highly mismatched N-rich GaN_{1-x}Sb_x alloys

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In order to effectively convert solar energy into electric power or any other form of usable energy we need to develop novel semiconductor materials, which will absorb over the full solar energy spectrum. Our research concentrates on GaN materials alloyed with group V anions of very different size and electronegativity, the so called highly mismatched alloys (HMAs). The electronic structure of the conduction and valence bands of HMAs can be well described well by the band anticrossing (BAC) model. For example, the BAC model predicts an energy gap ranging from 0.7eV to 3.4eV for GaN_{1-x}As_x alloys. An even stronger modification of the band structures is anticipated for the GaN_{1-x}Sb_x and GaN_{1-x}Bi_x alloys since the mismatch in the anions is more extreme. The large band gap range and controllable conduction and valence band positions of these HMAs make them promising materials for efficient solar energy conversion devices. However, the synthesis of these alloys is difficult due to the large size differences between the anions.

Recently we succeeded in growing GaN_{1-x}As_x and GaN_{1-x}Bi_x alloys over a large composition range by plasma-assisted molecular beam epitaxy (PA-MBE). The enhanced incorporation of As and Bi were achieved by growing the layers at extremely low growth temperatures. Although GaN_{1-x}As_x alloys become amorphous for x>0.1, optical absorption measurements show a progressive shift of the optical band gap to lower energy, from ~3.4eV to ~0.8eV, with increasing As content. The results strongly suggest that amorphous GaN_{1-x}As_x alloys have short-range order resembling random crystalline GaN_{1-x}As_x alloys. The large band gap range of the amorphous GaN_{1-x}As_x covers much of the solar spectrum making this material system a good candidate for full spectrum multi-junction solar cells. The amorphous nature of the GaN_{1-x}As_x alloys is particularly advantageous since low cost substrates such as glass can be used for solar cell fabrication. To the best of our knowledge, there is currently no information available on the growth of N-rich GaN_{1-x}Sb_x alloys.

In the current presentation we will discuss our first results on MBE growth and properties of N-rich GaN_{1-x}Sb_x alloys. We have grown layers on sapphire substrates at different temperatures, under a wide range of Sb fluxes and under different Ga to N ratios. Due to the large difference between N and Sb, the GaN_{1-x}Sb_x alloys become amorphous at Sb concentrations above x~0.05. Although GaN_{1-x}Sb_x alloys become amorphous, optical transmission and reflectance measurements showed strong absorption at ~1eV for GaN_{1-x}Sb_x layers. Differences and similarities between MBE growth conditions for GaN_{1-x}As_x, GaN_{1-x}Bi_x and GaN_{1-x}Sb_x alloys will be discussed and structural, optical and electrical properties of these highly mismatched alloys will be compared.

In-situ RF-MBE growth of AlO_x/n-GaN composite structures

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We have been developing an in-situ MBE growth technique to grow III-oxide thin films directly onto nitride semiconductors in an ultra-high-vacuum chamber to improve the device characteristics of GaN HFETs and to make novel device structures. Using this technique, we don't need to worry about surface oxidation that affects the formation of surface (interface) states and we should be able to form ideal oxide/nitride interfaces [1-3]. In this paper, we report on in-situ RF-MBE growth of AlO_x/n-GaN composite structures in the same growth chamber without exposure of the nitride surface to the atmosphere.

We used an MBE machine equipped with nitrogen and oxygen RF-plasma cells in the same growth chamber. This configuration enables us to grow nitride/oxide composite structures without exposure of the sample surface to the atmosphere. First, we grew nitride structures on c-plane sapphire substrates. After nitridation of the substrate surface, AlN buffer (300 nm), unintentionally doped GaN (500 nm), and Si-doped GaN (1000 nm) layers were grown on the substrate in sequence. The substrate temperatures (T_S) for the AlN and GaN layers were 900 and 750°C, respectively. The Si-doping concentration was $2.5 \times 10^{17} \text{ cm}^{-3}$. After growth of the nitride structure and cooling of the substrate, the sample was transferred to a buffer chamber connected to the growth chamber and kept in an ultra-high-vacuum atmosphere. After that, the substrate was transferred to the growth chamber again, and an AlO_x thin film was grown on the n-GaN in the following sequence. First, a 3-nm-thick Al was deposited on the n-GaN at $T_S=200^\circ\text{C}$ by using the same K-cell for the AlN growth. Then, the Al was oxidized by O-plasma irradiation for 5 min. Two samples were fabricated: one sample was taken out from the chamber just after the oxidation (sample A), and the other was heated up to $T_S=800^\circ\text{C}$ and annealed for 10 min (sample B). The structural properties of both samples were characterized by atomic force microscopy (AFM) and x-ray photoelectron spectroscopy (XPS).

Surface AFM images showed that coverage of the AlO_x film on the n-GaN was much improved by the annealing at 800°C. The Al 2p XPS spectrum of sample A had two peaks related to Al-O bonding and metal Al. This was because part of the deposited Al remained as metal due to insufficient oxidation at a substrate temperature of 200°C. On the other hand, there was no metal Al peak from sample B, indicating that the remaining metal Al transformed into AlO_x during the annealing at $T_S=800^\circ\text{C}$. The Ga 3d spectrum from sample A showed a small Ga-O peak due to oxidation of the n-GaN surface, whereas the spectrum of sample B showed no such peak. This result is consistent with the AFM observation. These structural properties indicate that a high-quality AlO_x thin film and AlO_x/GaN interface with good surface coverage were formed by the process of sample B, that is, by the three-step process of (1) Al deposition at low temperature, (2) oxidation by O plasma irradiation at the low temperature, and (3) annealing at high temperature.

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Nano-scale correlation of structural and optical properties in Group-III-Nitrides by (S)TEM-cathodoluminescence spectroscopy

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For a detailed understanding of complex semiconductor heterostructures and the physics of devices based on them, a systematic determination and correlation of the structural, chemical, electronic, and optical properties on a nanometer scale is essential. Luminescence techniques belong to the most sensitive, non-destructive methods of semiconductor research. The combination of luminescence spectroscopy – in particular at liquid He temperatures - with the high spatial resolution of a scanning transmission electron microscopy (STEM) ($\delta x < 1$ nm at RT, $\delta x < 5$ nm at 10 K), as realized by the technique of low temperature scanning transmission electron microscopy cathodoluminescence microscopy (STEM-CL), provides a unique, extremely powerful tool for the optical nano-characterization of semiconductors, their heterostructures as well as their interfaces.

Our CL-detection unit is integrated in a FEI STEM Tecnai F20 equipped with a liquid helium stage ($T = 10\text{K} / 300\text{K}$) and a light collecting parabolic mirror. Panchromatic as well as spectrally resolved (grating monochromator) CL imaging is used. In CL-imaging mode the CL-intensity is collected simultaneously to the STEM signal - typically chemical sensitive HAADF Z-contrast - at each pixel. The TEM acceleration voltage is optimized to minimize sample damage and prevent luminescence degeneration under electron beam excitation. In CL-spectral imaging mode, a complete CL spectrum is recorded at every single pixel. Subsequently, by evaluating the complex data matrix $I_{\text{CL}}(\lambda, x, y)$, sets of simultaneously recorded monochromatic mappings $I_{\text{CL}}(\lambda_i, x, y)$, CL peak wavelength mappings $\lambda_{\text{CL}}(x, y)$, local spectrum linescans, local CL spectra, etc. can be processed.

Typical results which will be presented include nm-scale correlation of the optical properties and strength and appearance of structural defects in: strain engineering AlN interlayers in GaN-on-Si structures, lattice matched AlInN/GaN distributed Bragg reflectors (DBRs) and complete vertical cavity surface emitting laser VCSEL structures with all-epitaxial as well as hybrid DBRs for blue (InGaN QWL cavity) and UV (GaN cavity) emission.

Minority carrier diffusion lengths of $\lambda < 17$ nm on one hand, as well as the efficient carrier transfer over > 150 nm into the quantum wells are directly measured in STEM-CL linescans. The impact of structural defects like dislocations is directly visible. The individual $\lambda/4$ layers of the epitaxial DBRs and the nanoscale properties of their mutual interfaces are clearly resolved.

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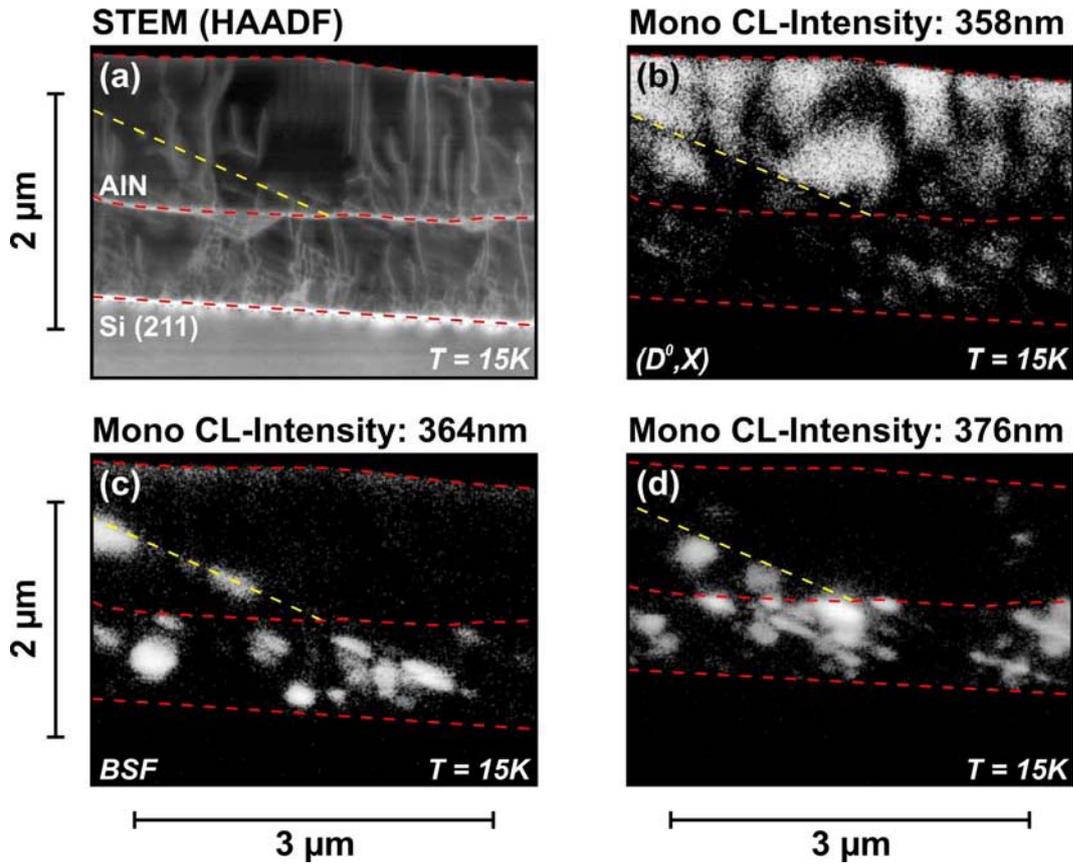


Fig. 1: Semipolar GaN grown on Si(211) substrate: STEM (a) and monochromatic TEM-CL images of the log. intensity of (D^0, X) band edge luminescence (b), basal plane stacking fault related luminescence (c) and donor acceptor pair band or prismatic stacking fault related luminescence (d), demonstrating the impact of an AlN interlayer.

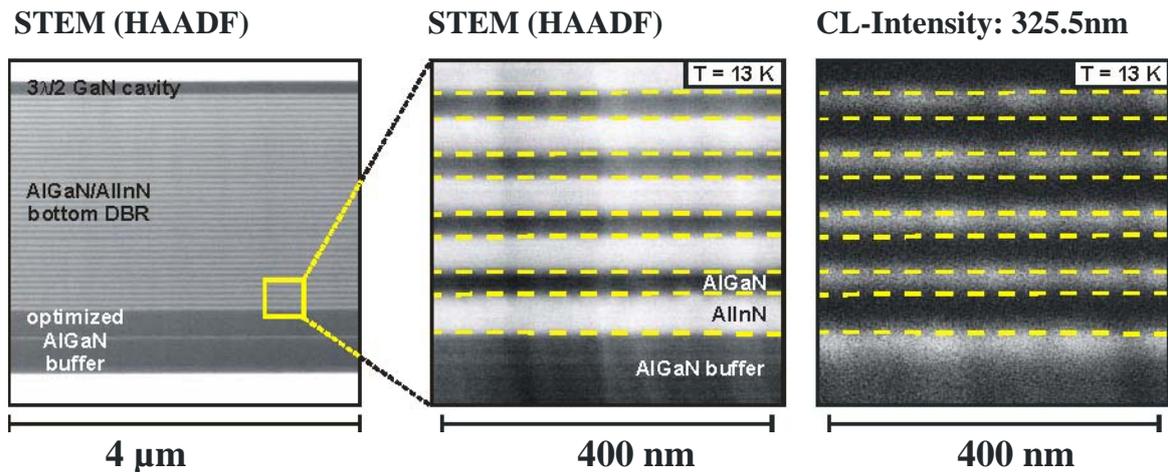


Fig. 2: $3\lambda/2$ GaN bulk cavity on 35 pair epitaxial AlGaInN DBR.

EPR and ODMR defect control in AlN bulk crystals

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The results of electron paramagnetic resonance (EPR) studies of shallow donors and deep level color centers in bulk AlN crystals are presented. AlN bulk crystals were grown by the sublimation sandwich method in a resistively heated furnace with axially symmetric graphite heating elements. A key feature of this growth technique is the use of a pre-treated tantalum growth crucible, which was pre-carbonized [1]. Two shallow donors (presumably oxygen located on the nitrogen site and carbon located on the aluminum site) are suggested to exhibit self-compensation due to the DX-relaxation [2]. Third shallow donor (presumably silicon on the Al site) shows the shallow donor behavior up to the room temperature and provides a reliable *n*-type conductivity at room temperature. The site identification was based on comparisons of the EPR linewidths, whose magnitude is mainly determined by the hyperfine interaction with ²⁷Al. The values of the Bohr radius for SDs in N and Al positions were estimated from the EPR linewidths to be ~10 Å and ~20 Å, respectively.

The investigations revealed the presence of deep-level transition metals impurities in bulk AlN crystals: Fe²⁺ (spin S=2) and some paramagnetic centers with S=3/2, we suggest Cr³⁺ and Ni³⁺ as the possible candidates. The EPR spectra of Fe²⁺ were observed up to the room temperature.

Joint studies of the EPR, electron-nuclear double resonance (ENDOR), optical absorption and thermoluminescence induced by X-ray irradiation allowed establishing the structure of the deep level color centers in AlN - a neutral nitrogen vacancy V_N⁰ with an optical absorption band in the visible region with a maximum at ~450 nm and the energy level ~0.75 eV relatively to the AlN conduction band. The yellow-brown coloration of crystalline AlN substrates was concluded to be due to the presence of neutral nitrogen vacancies. In contrast, the presence of vacancies in the ionized nonparamagnetic state does not lead to optical absorption in the visible region, that is, makes AlN crystal transparent.

Optically detected magnetic resonance (ODMR) was used to study defects and the spin-dependent recombination processes in AlN crystals. One of the defects was concluded from the ODMR spectrum orientation dependence to correspond to exchange-coupled pairs of nitrogen vacancies (N divacancy), which are in two adjacent (0001) planes of nitrogen. This conclusion was made on the basis of the angular dependencies of the ODMR spectra. AlN bulk crystals containing V_N⁰ color centers were shown to exhibit ODMR signals of the V_N⁰ defects, corresponding to a reduction in the luminescence intensity, as well as a resonance reduction in the luminescence intensity in zero magnetic field without microwave due to the existence of a spin-dependent nonradiative process, which shunted the luminescence channel when the external magnetic field is less than the internal magnetic field generated by the V_N⁰ hyperfine interaction.

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Atom Probe Tomography Characterization of InGaN Device Structures

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Thin-film light-emitting diodes (LEDs) composed of GaN/In_xGa_{1-x}N/GaN quantum well structures (QW) are integrated into modern optoelectronic devices because of a tunable InGaN bandgap which enables emission of the full visible spectrum [1,2]. A key issue related to ultra-efficient emission from these materials is compositional inhomogeneity. Distribution of indium within the QWs is central to understanding carrier localization and recombination properties affecting device performance; however, the measurement of QW compositional homogeneity is an ongoing challenge. Transmission electron microscopy (TEM) and atom probe tomography (APT) [3] are well suited for measuring In-composition distributions at the nanometer-scale, but each method has inherent limitations.

In this presentation, we detail the methodology used to assess APT data for compositional homogeneity, both in terms of large-scale (In-composition as function of depth within the film) and small-scale (nm-sized In-rich clustering), and detail some of the limitations of the technique [4,5]. Interfacial abruptness, compositional variation and the degree of In-clustering is reported for an off-the-shelf LED. We show that careful selection of acquisition conditions and analysis parameters are necessary to achieve accurate results.

Illustrations of synthetic data containing various degrees of clustering shows that use of non-statistical visualization techniques to evaluate In-rich clustering in these materials can be misleading. Ultimately, the dataset size, contrast of the indium within the matrix and the precipitate, and volume fraction of the precipitate region all affect the ability to resolve the presence of In-clustering. The technique also reveals an optimum bin size for a given fixed precipitate size with increased sensitivity for larger precipitate sizes.

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Observation of GaInN strain relaxation by *in situ* X-ray diffraction monitoring during metalorganic vapor phase epitaxy growth

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GaInN has aroused wide interest as a promising material for many applications, such as LEDs, laser diodes, sensors and solar cells. Owing to the lack of a homogenous substrate, a GaInN epilayer is typically grown on c-plane GaN. Because the crystallinity of GaInN/GaN rapidly decreases when it exceeds the critical film thickness, improving the performance of such devices is essential to understanding relaxation process of the GaInN/GaN heterostructure. Thus far, there have been many reports on the GaInN/GaN heterostructure. The characterizations of this heterostructure have mainly been carried out by *ex situ* measurement, such as by X-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). However, *in situ* observation techniques will enable us to understand the heterostructure relaxation process in more detail. In this study, we characterized the GaInN/GaN heterostructure on sapphire using a novel *in situ* XRD measurement system. We also discuss the relaxation process of the GaInN/GaN heterostructure.

Our *in situ* XRD system with a metalorganic vapor phase epitaxy reactor does not perform scanning. X-rays from the tube spread out as shown in the figure. These X-rays are focused using a Johansson curved crystal. By irradiating the focused X-rays on the substrate, this *in situ* XRD system can perform measurements without scanning the angle. In addition, the dispersed X-rays are detected using a one-dimensional CCD. The obtained configurations are measured similarly in only 0.1-1 s using a general XRD system. In this *in situ* XRD system, the crystalline (tilt) component and the distribution of the lattice constant c can be evaluated simultaneously by measuring the full width at half maximum (FWHM). Normally, FWHM decreases with increasing film thickness; however, this is not the case for thin film. The experimental and theoretical FWHMs were shown to similarly decrease with increasing Ga_{0.87}In_{0.13}N film thickness up to approximately 20 nm. However, the FWHMs of the Ga_{0.87}In_{0.13}N films with thicknesses exceeding 20 nm tended to deviate from the theoretical value. Moreover, the FWHM saturated when the film thickness exceeded approximately 100 nm. This result suggests that strain relaxation in Ga_{0.87}In_{0.13}N films is induced by increasing the Ga_{0.87}In_{0.13}N film thickness. Therefore, the estimated values of hc_1 and hc_2 are 20 nm and 100 nm, respectively. Because we suspect that the plastic relaxations in Ga_{0.87}In_{0.13}N occurs at approximately these thickness, we fabricated and characterized 10-, 35-, 80-, and 110-nm-thick Ga_{0.87}In_{0.13}N films on GaN. According to the results of *ex situ* TEM, SEM, and XRD analysis, the threading dislocations (TDs) almost reached the surfaces of these typical GaInN films grown on a GaN layer. We found that the Ga_{0.87}In_{0.13}N films formed surface pits at the ends of the TDs with increasing Ga_{0.87}In_{0.13}N film thickness. TDs appeared to laterally extend with increasing Ga_{0.87}In_{0.13}N film thickness. This suggests that TDs are bent during growth by in-plane compressive stress between Ga_{0.87}In_{0.13}N and GaN. Therefore, hc_1 is associated with bent TDs, which form growth pits at the ends of TDs. Moreover, we confirmed that misfit dislocations (MDs) were introduced when the thickness exceeded hc_2 . We consider that this finding will be very helpful for the growth of the high performance device structures.

EXAFS study of GaN/AlN multiple quantum wells grown by ammonia MBE

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GaN/AlN multiple quantum wells (MQW) are promising materials for novel optical applications. They serve as base materials for light emitting devices operating in ultraviolet spectral region, and high-speed infrared intersubband optical modulators and photodetectors. It is well known that the optical properties of nitride QWs are strongly affected by arrangement of heterointerfaces. In the present work a local structure of heterointerfaces in hexagonal QWs was investigated by extended X-ray absorption fine structure (EXAFS).

Hexagonal GaN/AlN MQW with 400 nm thick AlN buffer layer were grown by ammonia MBE on (0001)-oriented sapphire substrates. GaN well and AlN barrier thicknesses were varied from 1.0 to 4.0 monolayers, whereas the number of period ranges from 20 to 260. For preliminary characterization of MQW structures X-ray diffraction, transmission electron microscopy, Raman, photoluminescence measurements were performed. EXAFS spectra of the GaK - edges were measured at the VEPP-3 storage ring of the Budker Institute of Nuclear Physics (Novosibirsk).

Local arrangement of Ga atom were calculated using EXCURV-98 simulation tool. A coordination number for nitrogen atoms in the first coordination sphere of Ga atom were kept equal to 4. It was found that the coordination number for gallium atoms rises with increase the thickness of GaN layers. Moreover, a distance between Ga and N atoms, as well as the distance between Ga-Ga atoms agree well with that for a bulk GaN. The latter evidences in relaxation of GaN layers, which were usually in about two times thicker than the AlN barriers. It should be noted that we have found earlier that GaN quantum dots embedded in the AlN matrix are strained. The strain value in wells and barriers of MQW structures were evaluated by Raman spectroscopy and X-ray diffraction as well. Structural defects arising at relaxation of MQW were examined by transmission electron microscopy.

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Characterisation of Nitride Materials by Synchrotron X-ray Microdiffraction Reciprocal Space Mapping

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X-ray Reciprocal Space Mapping (RSM) is a powerful tool to explore the structure of semiconductor materials. However, conventional lab-based RSMs are usually measured in two dimensions (2D) ignoring the third dimension of diffraction-space volume. The idea of full three-dimensional (3D) RSM to obtain information about the structure of materials was first introduced by Fewster *et al* [1].

Recent advances in X-ray optics have led to the development of various micro- and nanofocusing optical elements [2]. On the other hand the progress in X-ray detector technology has led to availability of low-noise single photon counting 2D area detectors based on Complementary Metal–Oxide–Semiconductor (CMOS) hybrid-pixel technology. We report the use of a combination of X-ray microfocusing and state-of-the-art 2D area detectors to study the full volume of diffraction–space while probing the material on a microscale.

InGaN, InAlN epilayers and InGaN multiple Quantum Wells (MQW) grown on Epitaxial Lateral Overgrown (ELOG) GaN template were studied. Microdiffraction X-ray Reciprocal Space Mapping in 3D allowed us to gain invaluable information about the microstructure of nitride materials and heterostructures. It was found that “seed” InGaN mosaic nanocrystallites are *twisted* in respect to the ensemble average and *strain free*. This indicates that the growth of InGaN epilayers follows Volmer-Weber mechanism with nucleation of “seeds” on strain fields generated by the *a*-type dislocations which are responsible for the twist of underlying GaN mosaic blocks [3]. In the case of InAlN epilayers formation of composition gradient was observed at the beginning of the epitaxial growth. The utility of microdiffraction 3D RSM for the ELOG sample will be discussed.

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MOVPE GaN on Si for Solid-State Lighting Applications

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LED light bulbs will eventually replace incandescent bulbs for lighting our homes and offices: compact fluorescent lamps (CFLs) and fluorescent tubes are a temporary solution to energy efficient lighting. The main reason LEDs have not yet displaced CFLs and fluorescent tubes is the cost. For example a good quality 40W-equivalent LED retrofit bulb, which is warm white and dimmable, costs about US\$30, with 60W and 100W replacement LED bulbs being considerably more expensive.

LEDs grown on large-diameter Si substrates can provide a low-cost manufacturing route for GaN-based solid-state lighting. The estimated cost reduction is about a factor of five, due to Si being a cheaper substrate than sapphire or SiC and, in addition, growth on large-diameter substrates reducing the growth and processing costs per LED chip relative to growth on smaller-diameter substrates.

There are two main problems in growing GaN on large-area Si substrates. First, the thermal expansion of GaN is 50% higher than that of Si, so on cooling down from the growth temperature the GaN is in tension and cracks. This problem can be solved by introducing AlGaIn layers during the growth, which introduce compressive strain to counter the tensile strain in the GaN when the wafer is cooled. The second problem is the higher dislocation density (typically 10^{10} cm⁻²) when GaN is grown on Si due to the large lattice mismatch. This problem is solved by using dislocation reduction techniques during growth, for example by introducing a thin silicon nitride layer in the GaN.

Using the above techniques we have grown blue (450 nm) GaN multi-quantum-well structures on 6-inch Si substrates with a PL-IQE of 67% at room temperature. Our blue GaN LEDs on 6-inch Si substrates have a light power output of 50 mW at 350 mA with the Si substrate on: flip-chipping and Si substrate removal will significantly improve this result. We conclude that it is possible to grow GaN-based LEDs on 6-inch Si with high efficiency, comparable to the efficiency of LEDs grown on two-inch sapphire or SiC. The substantial manufacturing cost savings resulting from growth on large-area Si substrates will enable the widespread use of GaN LEDs in home and office lighting. We thank the Industrial Technology Research Institute (ITRI), Taiwan, for performing the processing.

Stimulated Formation of InGaN Quantum Dots by MOCVD

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Achievement of a high emission efficiency of GaN based light-emitting diodes (LEDs) in the whole visible optical range is one of the key tasks in modern optoelectronics due to a wide use of such LEDs for general lighting. One of the main problems limiting the efficiency of the III-N based LEDs is the high density of dislocations in the active region of a device containing epitaxially grown heterostructures typically composed of largely lattice-mismatched materials. This problem becomes particularly important for green-red LEDs containing InGaN layers with a high indium concentration. One of the promising ways to increase the efficiency of InGaN based LEDs is to replace the continuous two-dimensional InGaN layer by an array of isolated InGaN islands (quantum dots).

In this work different methods of stimulation of the InGaN quantum dot (QD) formation were investigated. Investigated heterostructures were grown by MOCVD. Structural properties of the grown samples were studied by atomic force microscopy (AFM) and high-resolution transmission electron microscopy (HRTEM). Geometric phase analysis (GPA) of HRTEM images was applied.

Effects of growth pressure and growth interruptions on the structural properties of the InGaN ultrathin layers were revealed. It was shown that increase in the growth pressure as well applying of the growth interruptions in mixtured nitrogen-hydrogen atmosphere stimulate phase separation inside InGaN ultrathin layers and lead to formation of the QDs. Except effect on the QD formation the growth interruptions resulted in transformation of the continuous InGaN layer to large isolated islands having sizes on several tens of nanometers. It was shown that applying of the interruptions during growth of the active region of LEDs modifies emission spectra and shifts maximum in the current dependence toward small currents.

Method for the fabrication of well separated InGaN islands based on growth of composite InGaN/GaN/InAlN structures was proposed. The idea of the method is based on a creation of preferential nucleation sites for these islands by the generation of 3D strain fields from InAlN islands lying beneath. InAlN islands are formed from a 2D In_{0.17}Al_{0.83}N (lattice-matched to GaN) layer following an InAlN alloy tendency for a strong phase separation. AFM studies of the structures created by a deposition of thin (4-20 nm-thick) InAlN layers have shown the presence of 3D islands having sizes of several tens of nanometers and density of $8-11 \times 10^{11} \text{ cm}^{-2}$. GPA of HRTEM images of the composite InAlN/GaN/InGaN heterostructures confirmed a 2D-3D transformation of the overgrown InAlN layers and demonstrated a strong phase separation there. A thin, continuous AlN layer lies beneath. 3D InAlN islands with a vertically increasing In composition are formed above. All islands are covered by a thin AlN layer at their summits. Isolated InGaN islands have been detected above the InAlN islands. A vertical correlation in the positions of the InGaN and InAlN islands is revealed. Composite InAlN/GaN/InGaN heterostructures with high content InGaN layer demonstrated emission in the range of 540-620 nm in dependence on the InAlN layer thickness.

Reactants injector temperature effect on III-Nitrides materials deposition in the high speed vertical rotating disc MOCVD reactor

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Cost of ownership reduction is one of the major goals for LED industry. Intensive research is focused on optimization of the process parameters in order to improve deposition process yield and throughput as well as quality of deposited materials using commercially available and research reactors. Less frequently those efforts are concentrated on optimization of the process reactor itself. In this presentation we will discuss process improvements that can be achieved by changing temperature of the reactant injector in the range of 50-200 C in the high speed vertical rotating disc MOCVD reactor (large scale production 465i MOCVD system manufactured by VEECO Instruments, Inc.).

Flow dynamic modeling demonstrated that increasing injector temperature allowed to achieve stable non-circulating flow regime in the reactor with up to 70% reduction of the shroud flow of H₂ and N₂. This also permits to perform deposition runs at pressure up to atmospheric which was prohibited before due to the high total flow required to support plug flow conditions. As was reported previously [1] the adjusted flow conditions allowed to achieve GaN deposition rates above 20 um/hour without sacrificing materials quality or deposition uniformity. Reduction of the heat flux through the carrier as a result of hotter injector leads to the reduced bow of the substrates which improves its temperature uniformity. We observed up to 50% bow reduction based on the measurement by the in-situ deflectometer. Smaller substrate deformation also results in the significant GaN uniformity improvement. Another advantage of the reduced heat flux is the lower dependence of the substrate temperature from the gap between substrate and the carrier. As any carrier and substrate have a dimensional variations, this feature provides for better substrate temperature uniformity.

We will also report on the effect of the increase injector temperature on particles formation and distribution, as well as changes in Mg doping efficiency.

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High-quality III-nitride templates with overgrown porous structure

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A technique for fabrication of high-quality III-nitride templates with overgrown porous structure for the GaN-based LEDs with improved material quality and light output is presented. The technique employs artificial micron scale voids located at the GaN-sapphire interface, which serve for reducing mechanical stress and dislocation density in the structures and improve the light scattering properties, thus enhancing light output from the LEDs.

When developing the technique, first, modeling was performed, which showed that formation of a regular porous structure in the GaN/sapphire template resulted in 20% reduction of mechanical stresses, with the value and localization of stresses being defined by the linear size of the voids. On the basis of the results of the modeling, the templates were fabricated via multi-stage Metal-Organic Chemical Vapor Deposition (MOCVD) growth of GaN layers on sapphire substrates with patterning of the first deposited GaN layer using standard photolithography process, metal masking and dry etching for forming hexagonal-like shaped voids ('pores') with various linear dimensions and inter-void spacing. The etched openings were overgrown by GaN layer with MOCVD using various III/V ratios. The templates obtained were studied using scanning electron microscopy (SEM) and X-ray diffraction (XRD).

It was established that varying the parameters of the initial mask pattern and growth conditions, the sidewall inclination angle of the voids could be controlled from nearly vertical (85°) to fully inclined (60°) for GaN layers with etched pore openings. Studies performed with the use of transmission electron microscopy (TEM) showed that void walls could effectively re-direct dislocations, thus reducing their density near the surface of the template. XRD data indicated substantial improvement of the crystalline quality of epitaxial GaN layers grown on the templates with inclination angle of the voids decreasing from 85° down to 60°.

A typical LED structure grown on the template would comprise a 1.5 μm thick Si-doped n-GaN layer, 5 pairs of InGaN/GaN quantum wells, a 50 nm thick un-doped GaN layer to prevent the desorption of In and back diffusion of Mg, and finalized with the growth of 200 nm thick p-GaN and an in-situ annealing. When comparing the performance of the structures with that of a reference LED grown on a conventional sapphire substrate, it appeared that at 20 mA current injection, the reference LED gave 9.4 mW of light output, while introducing voids at the GaN-sapphire interface enhanced the light output up 11.4 mW. The series resistance and reverse leakage current values for LEDs with ~60° inclined sidewall voids were much smaller as compared to the reference LED structure, which confirmed improved material quality of GaN grown on the templates, indicating a reduction in TDs which could act as current leakage channels. Integrating sphere measurements on LED chips showed a maximum of 21 % increase in the light output for the LEDs having the improved material quality, largest voids and inclined sidewalls. This confirms that high-quality III-nitride templates with overgrown porous structure show good prospects in III-nitride LED technology.

Three zone topside temperature control in a close coupled showerhead[®] MOVPE reactor

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For MOVPE production of GaN based LEDs control of the wafer surface temperature is critical to device yield whether this be homogeneity over the wafer area, the stability during a process run or the reproducibility run to run. Temperature is a main parameter for all steps in the process and in particular growth temperature of the InGaN quantum well dominates the emission wavelength of the devices as a result of the sensitivity of In content to temperature.

In this study a CRIUS[®] II-XL Close Coupled Showerhead[®] (CCS) MOVPE reactor with a 19x4" sapphire wafer configuration was used. The heating system incorporated 3 concentric heater zones to enable a flat temperature profile over the wafer area during the entire process. In conventional operation the power ratio of these three zones can be varied to optimise uniformity of each part of the LED process whilst the overall power is controlled by a thermocouple under the wafer carrier. In-situ topside measurements are required to fully optimise the entirety of a complex LED structure grown with multiple growth conditions and transients.

To monitor the temperature profile over the entire radius of the wafer carrier we developed the Argus topside pyrometer. This utilises an array of dual wavelength detectors over the radius of the wafer carrier, using the showerhead injectors as collimators, which after one revolution of the wafer carrier delivers a temperature map of the wafers. The surface temperature of each zone was calculated from the mapping and zones were independently closed loop controlled to follow the set-point temperature from the process recipe. The controller was configured to handle partial as well as full wafer loads.

To assess the reproducibility of control 5 consecutive fully loaded 19x4" LED process runs were performed, followed by 6 partially loaded runs each with 7 prime wafers and 12 recycled dummy wafers. The wafer carrier was exchanged between each run and three zone top side temperature control (ZTTC) was used for the entire process above 500°C. The wafers were evaluated by photoluminescence (PL) mapping, and white light interference thickness mapping with 2 mm edge exclusion.

Near optimum thickness uniformity values of σ (on-wafer) = 1.0% and σ (wafer-to-wafer) = 0.6%, and PL peak wavelength uniformity of σ (on-wafer) = 1.3 nm and σ (wafer-to-wafer) = 0.9 nm, were observed for all fully loaded runs. This demonstrates consistent control of all three heater zones.

The average PL peak wavelength of the five fully loaded runs was 452.1 nm with a range of run averages of 1.2 nm and the range of the entire 95 wafers was 5.5 nm. The 6 partial loaded runs had similar uniformities and only increased the full range of run average wavelengths to 1.7 nm; implying control of partial loads was also successful. These results clearly demonstrate that ZTTC can improve LED production yield.

Time-resolved luminescence studies on AlN and high AlN mole fraction AlGaN alloys

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For improving the internal quantum efficiency of deep ultraviolet (DUV) light emitters containing AlN or high AlN mole fraction (x) $\text{Al}_x\text{Ga}_{1-x}\text{N}$ active regions, quantitative understanding of radiative and nonradiative lifetimes is essential. However, there have been few reported results [1-4] on the recombination dynamics in AlN and $\text{Al}_x\text{Ga}_{1-x}\text{N}$ alloys, which exhibit the near-band-edge (NBE) emission peak shorter than 240 nm, due to limited availability for a desirable DUV femtosecond excitation source. In this presentation, the results of low-excitation DUV time-resolved photoluminescence (TRPL) and time-resolved cathodoluminescence (TRCL) measurements on AlN and high- x $\text{Al}_x\text{Ga}_{1-x}\text{N}$ epilayers of various crystal qualities will be shown to discuss the relationship between the lifetimes and crystal qualities, making connections with the results of positron annihilation spectroscopy (PAS) [5].

The samples were grown by low-pressure metalorganic vapor phase epitaxy on a c -plane Al_2O_3 substrate and on an m -plane freestanding GaN substrate [1-4]. To excite carriers, we used frequency-quadrupled (4ω : 197~200 nm) fs Al_2O_3 :Ti laser [2,3] and a home-made photoelectron gun (PE-gun) excited with a frequency-tripled (3ω) fs Al_2O_3 :Ti laser [3,4]. Monoenergetic positron beam line [5] was used to determine the Doppler-broadening S parameter for the annihilation γ -rays of e^-e^+ pairs, which is used as the measure of concentration or size of negatively charged cation-vacancy defects [6].

We will demonstrate extremely radiative nature of AlN, as gross radiative lifetimes (τ_R) for a free excitonic polariton and bound exciton emissions of high quality AlN are as short as 10 ps at 7 K [2]. Next we show that the value is elongated up to 530 ps with the increase in S [3], irrespective of threading dislocation density. Because S represents the concentration or size of Al-vacancy defects and low-temperature-grown AlN contained high concentration O, C, and Si, the result implies spatial separation of electron-hole pairs in the band-tail. A continuous decrease in radiative lifetime with temperature rise up to 200 K for heavily-doped AlN reveals a carrier release from the band-tail. The low-temperature τ_R for $\text{Al}_x\text{Ga}_{1-x}\text{N}$ films of high- x is longer than the values for epilayers of low- x $\text{Al}_x\text{Ga}_{1-x}\text{N}$, AlN, or GaN due to the contribution of bound and localized tail-states. However, τ_R shows little change with temperature rise, and the value is a few ns at 300 K. The results essentially indicate an excellent radiative performance, although the luminescence efficiency of $\text{Al}_x\text{Ga}_{1-x}\text{N}$ DUV light-emitting-diodes reported so far is limited by the short nonradiative lifetime and low light extraction efficiency.

The m -plane GaN wafer was supplied by Mitsubishi Chemical Corporation. This work was supported in part by NEDO programs under METI, Grant-in-Aids for Scientific Research Nos. 23656206 and 18069001 under MEXT, Japan, and AFOSR/AOARD Grant No. FA2386-11-1-4013 monitored by Dr. G. Jessen.

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Higher than 90% internal quantum efficiency of photoluminescence in GaN:Zn,Si

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Co-doping of GaN with silicon and zinc leads to bright light emission at a photon energy of about 2.9 eV. Such a co-doping has been used in early LED structures for creating blue light emission. However, after the “invention” of the InGaN quantum well, the Zn-related emission is no longer used in LEDs. New interest in this material could arise from the field of single photon emitters, and high internal quantum efficiency is a prerequisite in this case. Whereas the external quantum efficiency (EQE) can be determined by merely measuring the number of photons emitted by the LED, the determination of the absolute internal quantum efficiency (IQE) of electroluminescence or photoluminescence (PL) in wide-bandgap semiconductors remains a continuing challenge.

In this work, the optical properties of high-quality GaN co-doped with silicon and zinc were investigated with temperature-dependent, continuous-wave and time-resolved PL measurements. Two groups of GaN:Si,Zn samples with different concentrations of Zn (uncapped and capped with AlGaIn film), as well as GaN:Si samples with different concentrations of Si, were grown by metalorganic vapor phase epitaxy on *c*-plane sapphire substrates. The concentration of free electrons in the GaN:Si,Zn samples [$(0.5-1.4)\times 10^{19} \text{ cm}^{-3}$] was determined from the Hall effect and from analysis of the PL data. The concentration of the Zn_{Ga} acceptors in these samples (from 5×10^{17} to $1\times 10^{19} \text{ cm}^{-3}$) was estimated from PL measurements.

The IQE of PL was determined from the analysis of the dependences of the PL intensity on the excitation intensity and temperature, and the simulation of these dependences with a phenomenological model based on rate equations. The model reproduces an important phenomenon: the quenching of a recombination channel with a high IQE causes a rise in efficiency of all the other PL bands. This quenching can be achieved by increasing the temperature or excitation intensity. Quantitative analysis of this phenomenon allows one to determine reliably the absolute IQE of PL. The absolute IQE of the PL in GaN:Si,Zn exceeds 90%, with the largest contribution coming from the blue luminescence band associated with the Zn_{Ga} acceptor.

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Surface acoustic waves and elastic constants of InN epilayers determined by Brillouin scattering

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Group-III nitrides are attracting much research interest because of their potential applications in high-efficiency solar cells, optical emitters and detectors. Although much efforts have been made recently, InN is still the least understood of this group. Concerning elastic constants, several theoretical studies have been carried out using different approaches, but no study has been reported so far on InN by Brillouin scattering. The only experimental determination of the InN elastic constants was recently obtained from grazing-incidence X-ray scattering by Serrano *et al.* [1] Because of its close relation to other structural properties, the study of the elastic constants can provide valuable information about the characteristics of the crystal lattice.

This work presents the first Brillouin scattering study on InN. The studied samples are c- and m-face InN thin films grown on GaN/sapphire and γ -LiAlO₂, respectively. Because of the small band gap of InN, the experiments have been performed in the absorption region. Therefore, only surface acoustic waves (SAW) can be detected which provide indirect information of the bulk elastic constants. The Brillouin spectra are recorded in backscattering configuration for different azimuthal and sagittal angles on both samples. Azimuthal angular studies show the isotropy of the SAW velocity on the c-face thin film and the anisotropy of the m-face sample. The experimental results suggest that both samples have a different set of elastic constants.

To evaluate the elastic constants of InN, numerical simulations based on the Green's function formalism have been performed and compared to the experimental results. A complete set of elastic constants is then proposed for both the m- and the c-face samples, respectively. The v_{SAW} angular dependences have been calculated using these elastic constants which describe satisfactorily the experimental results. The slightly different elastic properties of the studied samples may be caused by the structural defect anisotropy of InN non-polar surfaces.

This study demonstrates that Brillouin scattering is a powerful technique to obtain accurate information on the elastic properties, even in opaque materials such as InN.

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Splitting of surface-related phonon modes in Raman spectra of self-assembled GaN nanowires

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Self-assembled nanowires (NWs) currently are a subject of sustained interest in the scientific community motivated by both their potential applications and the new fundamental questions opened by these strongly anisotropic nano-objects. In this context, Raman spectroscopy has been increasingly used to study NWs and several new phenomena have been reported to date with respect to one-dimensional structures.

In this work, we report the observation and the detailed analysis of an unusual double peak structure in the Raman spectra of GaN NWs grown by Plasma-Assisted Molecular Beam Epitaxy (PA-MBE), which has been convincingly attributed to the Raman scattering by two surface related phonons. This assignment is justified by the experimental evidence of the frequency dependence of these surface phonons on the dielectric constant of the exterior medium. To interpret these experimental data, we used a theoretical approach which considers nanostructure ensembles as a homogeneous material. The interaction between NWs and the electromagnetic wave is addressed through the definition of an appropriate effective dielectric function, i.e. air-GaN effective medium model. The results of those numerical simulations are in good agreement with these experimental observations. Combined with additional experimental data obtained as a function of the laser polarization and the numerical aperture of the microscope objectives used to focus the laser on the sample, those calculations confirm that these two peaks can be respectively attributed to axial and planar surface-related phonon modes. It is shown that these two modes result from the anticrossing of two distinct surface phonon branches, occurring at the peculiar case $f = 0.4$, f standing for the filling factor value measured in this sample independently by SEM and AFM techniques. We show that this unusual double peak structure can be viewed as a clear manifestation of the splitting of the surface mode, leading to transverse and longitudinal components. Such effects should also be observed in all polar semiconductors with hexagonal wurtzite structure, particularly in materials for which the Fr  hlich electron-phonon coupling constant is high. By monitoring the shape and density of these 1D nanostructures, Raman scattering by those surface-related elementary excitations could be used as a fine probe of dielectric environment and of its anisotropy.

Linear and nonlinear optical investigations of periodic polarity-inverted GaN waveguides

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As a novel device application to the quantum optics, the periodic polarity-inverted GaN waveguides with periods of 2.0~50 μm were fabricated utilizing molecular beam epitaxy and electron beam lithography. Here in addition to the successful periodic reversal of the crystallographic orientations as confirmed by a piezoelectric force microscopy, grating structures were also formed spontaneously on the surface, which gives the occurrence of the characteristic resonances between incident light and guided modes. In this work, firstly basic optical properties were investigated by variable-angle optical reflectance measurements, in order to assign the origin of all these resonances. Next the configurations of wavelength conversions were discussed, taking into account their linear optical properties. Finally the demonstration on the enhanced second harmonic generation (SHG) was presented using a tunable laser as a fundamental wave.

Sample was mounted on the stage of a homemade optical goniometer with multiple scanning axes of polar incident angle θ , polar detection angle ζ and azimuthal angle ϕ , which is the in-plane angle between the plane of incidence and the Bragg vector of the grating. Both angles of θ and ζ were synchronously scanned, keeping the relationship of $\zeta = \pi - 2\theta$, which means the monitoring of the specular reflections. A white light from a xenon lamp was focused onto the patterned area with a period of 2.0 μm . The optical polarization was chosen by a Gran-Thomson prism. Then the reflected beams were collected by an optical fiber which was connected with CCD monochrometer with a sensitivity covering 300 through 1600 nm. In the actual sequence of measurements, the detection angle ζ dependence of the reflectance spectra, scanned from 15.0° to 150.0° with a step of 1.0°, were recorded. In addition to the optical interference fringes, clear reflectance dips originated from the resonance between the incident light and guided modes appeared, aside from a weak resonant feature due to the coupling of the diffracted light to the evanescent mode on the surface, known as Wood's anomalies. Taking into account the refractive index dispersions and the zone-folding effects invoked by the grating, origins of all the resonant features are successfully elucidated. Especially in case of resonant coupling to the guided modes, the corresponding orders of both the grating diffractions and the guided modes are assigned correctly.

Then the incident light was replaced by a p -polarized beam from a mode-locked Ti:sapphire laser with a pulse width of 80 fs and repetition rate of 81 MHz. Violet emission was collected by the identical optics used for the above reflectance measurements and dispersed through a grating monochrometer, and detected by a photomultiplier tube. In case of fundamental wavelength of 840 nm, the successful realization of SHG has been confirmed in terms of three evidences: a characteristic spectral shape with a peak wavelength of 420 nm which coincides well with that of the square of the fundamental wave, the power of violet emission which is proportional to the square of fundamental power, and the resonant enhancement of violet emission depending on the angular configuration which is characteristic to the grating coupling.

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Carrier dynamics in semipolar GaN / Al_{0.5}Ga_{0.5}N (11-22) nanostructures

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In the last few years, research on III-nitride ultraviolet light emitters has gained an increased interest. However, one of the major difficulties for the realization of these devices is related to the presence of a strong internal electric field that limits the optical properties of (0001) oriented polar materials. Due to the wurtzite structure with a unique polar axis and to the lattice mismatch, the polarization discontinuity at the interfaces between two materials generates electric field discontinuities which induce a red-shift of the luminescence and a strong decrease of the radiative transition rate known as the quantum confined Stark effect. So it is challenging to reach the UV range. An alternative approach to overcome this problem is the growth along a semipolar (11-22) orientation (which makes an angle of 58° with the (0001) orientation) which is currently being explored [1,2].

The semipolar structures started by the growth by metalorganic vapour phase epitaxy of 2 µm thick semipolar (11-22) GaN templates on m-plane sapphire substrates. Then the growth was performed by molecular beam epitaxy using ammonia as nitrogen precursor. 100 nm AlN were grown followed by an 800 nm Al_{0.5}Ga_{0.5}N thick layer. For the active region, 3 GaN semipolar nanostructures planes separated by 30 nm Al_{0.5}Ga_{0.5}N barriers were deposited and a fourth nanostructure plane was grown at the surface. The semipolar nanostructures are aligned and elongated along [1-100] [2].

By using semipolar GaN nanostructures, intense room temperature (RT) UV luminescence can be obtained on (1-100) sapphire. In that case, the internal electric field effects can be drastically reduced and as a consequence a shift to higher energies is observed compared to the polar case (i.e. quantum dots grown along (0001)). Also, compared to polar QDs with similar height, an increase of the room temperature radiative efficiency has been obtained. In order to assess the use of GaN semipolar nanostructures as active media for the realization of UV emitters, we present a detailed analysis of the optical properties and compare them with polar QDs. Continuous wave photoluminescence and time-resolved photoluminescence results are presented and analysed in correlation with high resolution transmission electronic microscopy studies. In particular, using different methods, we can estimate the value of the internal electric field inside the semipolar GaN/ Al_{0.5}Ga_{0.5}N nanostructures.

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Growth of quantum wire III-N nanostructures

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In spite of the huge interest for III-nitride nanowires (NWs), which is triggered by their exceptional structural and optical properties, the mechanism of nucleation of these nano-objects still remains obscure. In this context, we will first discuss the nucleation mechanisms of GaN NWs grown on Si(111) by molecular beam epitaxy. We will show that it is governed by the formation of 3D islands which have to reach a critical size before relaxing their elastic strain and next turning into NWs [1]. It will be demonstrated that these NWs exhibit a N-polarity [2].

Next, we will address the issue of InGaN nanowires, which are considered today as a promising material in the field of visible light-emitting diodes: the presence of free surfaces, intrinsic to nanowires, is expected to favor the strain relaxation and extend therefore the In composition range available, leading to green and yellow emission.

The InGaN nanowires have been grown by Plasma-Assisted MBE on Si(111). They consisted of a GaN NW base having a typical diameter of 30nm covered with a thick InGaN part on the top, about 30-70 nm long. The nominal In composition was varied between 0 and 0.5 by changing the In flux [3]. Based on SEM and HRTEM studies, it has been found that the morphology of the InGaN section was multiple, corresponding to several modes of elastic strain relaxation: plastic, elastic, or a combination of both, depending on In content and III/V ratio value. A general growth model of InGaN NWs will be presented, with a special attention paid to the purely elastic case, which is characterized by the spontaneous formation of InGaN/GaN core/shell heterostructures. This feature has been assigned to strain-assisted Ga and In diffusion on top of single InGaN quantum dots spontaneously formed on the GaN section. The observation of In clustering correlated to strain relaxation process will be discussed. Superlattices of InGaN single dots-in-GaN NWs have been grown. EDX analyses have revealed In inhomogeneities between successive dots but also along the growth direction within each dot, which is attributed to compositional pulling. By further performing spatially resolved cathodoluminescence (CL) experiments in a Scanning Transmission Electron Microscope (STEM) with an home-made high throughput CL detector, luminescence of single dots has been probed allowing one to put in evidence an enhanced luminescence from the high-In content top part with respect to the lower-In-content dot base [4].

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Single-crystal GaN on SiO₂ for monolithic integration with CMOS

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As the technology of GaN on sapphire enters into a mature phase, there has been much anticipation regarding the feasibility of monolithic integration of GaN with silicon. In spite of the impressive progress in demonstrating high-performance GaN LEDs and transistors on Si(111) substrates, fundamental issues such as lattice and thermal expansion mismatches remain unaddressed. Ideally one would like to build devices in a “GaN-on-insulator” configuration on Si(001) wafers to eliminate leakage paths and retain crystallographic compatibility with CMOS circuitry.

We report in this talk a novel process termed confined lateral overgrowth (CLO) of GaN out of a polycrystalline yet highly textured seed to produce single-crystalline GaN; the entire process is carried out on SiO₂ on standard Si(001) substrates. The CLO process is based on a mechanism termed *evolutionary selection* [1] that was proposed to explain the microstructural morphology in polycrystalline deposition, where the fastest-growing grains overtake their slower neighbors and become dominant at the growth front, thus creating a spontaneous filter that blocks misaligned grains so polycrystalline GaN can be laterally transformed into nearly mono-crystalline layer.

We first sputtered fibrously textured thin films on 500 nm of SiO₂ on standard Si(001) substrates having a desirable preferred orientation such as (0001) for AlN. This (sputtered) thin film provides a substrate-insensitive out-of-plane alignment yet with no in-plane alignment. To facilitate the CLO process, we fabricated constricted growth channels, grooves, and tunnels with one end adjacent to the thin-film seed, so the subsequent CLO process will “draw” GaN from the side of the sputtered thin film. The grains that are at the edge of the original patterned thin film now serve as the nuclei for the lateral overgrowth. The growth competition among GaN grains will favor those whose fastest-growing axes are aligned with the direction of lateral growth, and create a lateral orientation selection process that naturally improves the in-plane crystallographic alignment within a length of 2 to 4 μm. The consecutive orientation selection processes, namely the thin-film sputtering and the CLO applied along two orthogonal axes, are effective in producing quasi-monocrystalline AlGaInN layers for device integration. Using the principle described here, we have prepared single-crystalline GaN on SiO₂ on Si(001) substrates. The area of GaN is defined by the constriction and can be easily extended into device compatibility. The principle of lateral orientation selection is verified by electron back-scattering diffraction (EBSD) mapping, the crystallinity is affirmed by clear and high-order Kikuchi bands in EBSD. This process can be used to provide universal integration of GaN with oxide, metal, ceramics/glass, or other kind of substrates and can be extended to other material systems. We will discuss detailed fabrication, nanoscale material characterization, and device results at the conference.

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MOVPE growth of $\text{In}_x\text{Ga}_{1-x}\text{N}$ ($x\sim 0.5$) on Si(111) substrates with a pn junction on the surface

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An $\text{In}_x\text{Ga}_{1-x}\text{N}$ ($E_g\sim 1.8$ eV)/Si system is a promising combination for 2-junction tandem solar cell, because the current matching is achieved between the two subcells, which will result in a power conversion efficiency higher than 30% [1]. Furthermore, a low resistance ohmic contact predicted at the n- $\text{In}_{0.45}\text{Ga}_{0.55}\text{N}/\text{p-Si}$ interface is another advantage for the combination [2]. This means that no additional tunnel junction is necessary to connect the top and bottom cells. Although GaN and InN layers have been grown on Si substrate, the growth of $\text{In}_x\text{Ga}_{1-x}\text{N}$ with $x>0.1$ on Si remains a challenging task. Recently, MBE growth of $\text{In}_x\text{Ga}_{1-x}\text{N}$ with x up to ~ 0.3 has been reported on Si (111) [3]. However, there have been no reports on the MOVPE growth of InGaN on Si especially with an intermediate In content suitable to be paired with Si in tandem cell. In this work, we report the MOVPE growth of $\text{In}_x\text{Ga}_{1-x}\text{N}$ ($x\sim 0.5$) on Si(111) substrates with a pn junction on the surface.

The growth of $\text{In}_x\text{Ga}_{1-x}\text{N}$ is carried out using MOVPE with a horizontal reactor at a temperature range of 600~800°C in the pressure of 150 Torr. Two types of Si substrate are used; one is p-type Si(111) and the other is n-type Si(111) with a pn junction on the surface. The pn junction is formed by the B ion implantation into the n-type Si(111) substrate followed by the annealing at 1000°C. A 100nm thick AlN layer is used as an interlayer. A GaN/ $\alpha\text{-Al}_2\text{O}_3$ (0001) template is also employed as a substrate for comparison. By optimizing growth temperature and TMI/(TMI+TEG) molar ratio, single crystalline InGaN films with an In content around 0.5 are successfully grown on all the substrates without phase separation and metallic In incorporation. No significant difference is found in the FWHM of X-ray rocking curve (XRC) for the InGaN (0002) among the three kinds of substrate, indicating that the B ion implantation does not become obstacle of the epitaxial growth of InGaN. The FWHM of XRC is remarkably increased when the In content in InGaN is increased more than 0.3. The ohmic behavior is confirmed in the I-V characteristics for the n-InGaN/p-Si systems. It is found that the resistance between n-InGaN and p-Si is markedly decreased with increasing In content in InGaN film, in spite of the presence of the AlN interlayer.

Thus, we have successfully fabricated the high quality n- $\text{In}_{0.5}\text{Ga}_{0.5}\text{N}/\text{pn-Si}(111)$ hetero-structures for 2-junction tandem cell. Since we already reported the achievements of p-type conduction in InGaN on $\alpha\text{-Al}_2\text{O}_3$ (0001) up to an In content of 0.4 [4], these achievements will soon allow us towards the fabrication of InGaN-Si tandem cell. This work is supported in part by “Creative research for clean energy generation using solar energy” project in Core Research for Evolutional Science and Technology (CREST) programs of Japan Science and Technology Agency (JST).

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AlGaIn/GaN Heterostructures on Silicon (111) Substrates via Rare Earth Oxide Buffer Layers

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Growth of GaN on silicon substrates recently has been of great interest due to increased demand in high power, high voltage applications in hybrid cars and circuits in solar panels. The growth of GaN for HEMT applications is typically performed on silicon substrates to make use of the good thermal properties compared to that of sapphire which has traditionally been the substrate of choice for LED applications. However, large lattice mismatch and thermal expansion coefficient mismatch between GaN and silicon usually results in material quality issues which in turn degrades device performance. Moreover the GaN buffer layer thickness requirements scale with the operating voltage of the devices and therefore thicker GaN buffers are required as operating voltages increase. Growth of thick GaN on large diameter silicon wafers causes severe cracking and wafer bowing causing difficulties in further device processing.

In this paper we present the growth of GaN on silicon substrates using rare earth oxide, - Gd₂O₃, buffer layers. Growth of rare earth oxide buffer layers on silicon (111) substrates occur epitaxially due to a unique alignment of lattice constant of silicon and Gd₂O₃ in this case ($2a_{Si}=a_{Gd2O3}$). Growth of 30 nm thick Gd₂O₃ layers were performed in a Veeco Gen 20 reactor. Gadolinium was supplied to the surface by evaporation in a high temperature Knudsen cell; oxygen was supplied to the surface from a nozzle as molecular species. Growth of the Gd₂O₃ layer was monitored in situ using RHEED. Wafers are transferred to a Gen 3 reactor for further growth of GaN using a standard plasma source and Knudsen cell for gallium. Wafers are characterized ex-situ by AFM, XRD, TEM. Atomic force microscope investigation of GaN on Gd₂O₃ buffer layers show surfaces with 0.8 nm RMS roughness. Analysis of the growth layers by TEM show sharp interfaces without any interfacial layers. The material quality of GaN on Gd₂O₃ buffers are as good as GaN on silicon layers which suggest the suitability of these heterostructures for device applications.

Wafer curvature measurements performed on whole 100mm Gd₂O₃/Si wafers also indicate that the wafer becomes domed due to the difference in thermal expansion coefficients between silicon and the oxide layer upon cooling. Further growth of GaN on such a wafer counteracts the bowing effect and potentially can mitigate the common bowing problem observed in GaN on silicon epitaxy.

Growth of AlGaIn/GaN heterostructures on GaN/Gd₂O₃/Si buffers are also performed in order to achieve 2DEG channels for device applications. These structures are expected to benefit from the insulating properties of Gd₂O₃ ($E_g=5.0eV$) and in turn result in increased performance of HEMT structures for a given thickness of GaN. Results for electrical characterization will be presented at the time of the conference.

Growth of GaN-based heterostructures on silicon substrates

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After many years of research, the quality of GaN layers grown on silicon substrates has reached the standard quality of GaN layers grown on sapphire. Indeed, the threading dislocation density in best GaN layers grown on silicon could be as low as $5 \times 10^8 \text{ cm}^{-2}$ [1-3] which is a typical value for GaN layers grown on sapphire [4]. However, because of the large thermal expansion coefficient between GaN and Si, the growth of state of the art GaN layers on silicon substrates is more complicated than on sapphire. Indeed, in contrast to what is usually carried out on sapphire, more or less thick and more or less complicated (Al,Ga)N stacks need to be introduced in buffer layers in order to get crack-free GaN on silicon substrates. However, despite more challenging growth procedures on silicon substrates, there are so many advantages to grow on silicon that it is worth trying. Actually, the question whether the silicon will displace sapphire is becoming pertinent. If the answer to this question is still a matter of point of view, there is no doubt that the answer will be positive provided that growth schemes on silicon substrates get simpler.

This presentation will first discuss the important parameters that have to be taken into account for growing high-quality GaN layers on silicon substrates, highlighting the difficulties and solutions to overcome them. Then, the properties of state of the art GaN layers grown on silicon will be presented. Several heterostructures as well as devices taking advantage of the use of silicon substrates will be shown. Finally, the issue of the monolithic integration of GaN-based devices with silicon-based electronics will be discussed.

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Luminescence dynamics in undoped and Mg-doped III-Nitrides

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The recent development of applications of GaN and related materials in light emitting devices has been accompanied by intense studies on the physical properties governing the radiative processes in these materials. An understanding of the basic optical properties and how these are influenced by doping and defects in the materials is the basis for further developments.

Strain-free bulk GaN material of reasonably high purity ($< 10^{16} \text{ cm}^{-3}$) has been available only recently, allowing optical studies of intrinsic as well as doping related excitonic processes at near bandgap energies in GaN [1]. These studies have revealed details of the bound exciton electronic structure as well as the characteristic decay times related to the oscillator strength of these transitions. The results are influenced by near surface processes as well as exciton transfer to defect states at lower energies [2].

The p-doping is often a bottleneck in GaN based light emitters, and many studies have focused on revealing the connection between Mg-doping and the optical spectra of GaN, which turned out to be quite complex [3]. Recent work on homo-epitaxial Mg doped layers, both polar and non-polar, has revealed the creation of structural defects in such materials, initiated by the Mg-doping itself [4]. This helps to explain the unusual presence of several spectral features in Mg-doped GaN. We have studied photoluminescence spectra in GaN grown by MOVPE, HVPE and MBE, and will attempt an explanation of the various spectral features observed.

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Direct measurement of the Mg lattice site location in group III-Nitrides

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With respect to technological applications, Mg is so far the most feasible *p*-type dopant for III-nitrides semiconductors. However, this element will only be active as an acceptor if it substitutes for a group-III element, while Mg in other lattice locations will be electrically inactive or might even result in self-compensation, e.g. acting as an interstitial (double) donor. Despite its widespread use in nitride devices, very little direct information on the possible lattice sites of Mg exists. On the other hand, the Mg activation remains to this day a challenge, since (i) the III-nitrides are intrinsically *n*-type, (ii) the acceptor energy levels created by Mg are quite deep and (iii) its solubility is limited.

Using the emission channeling method [1] we have for the first time directly and unambiguously determined the lattice location of radioactive ²⁷Mg implanted in GaN, AlN and InN. Due to the short half life of this isotope ($t_{1/2} = 9$ min), the experiments were performed on-line at the ISOLDE facility at CERN. As was to be expected, the majority of ²⁷Mg was found in substitutional sites of Ga, Al and In, respectively. However a significant fraction of Mg was also identified close to the octahedral interstitial O sites of GaN and AlN. The most likely position of ²⁷Mg interstitials was determined, by means of comparing the experimental emission channeling patterns to the simulation results. These simulations represent a superposition of ²⁷Mg on the cation sites and on interstitial sites displaced from the ideal O sites along the *c*-axis.

The lattice location experiments were performed for several implantation temperatures. Both in GaN and AlN the interstitial Mg was converted to substitutional Ga and Al sites, respectively, at temperatures approximately around 600°C. By making reasonable assumptions about the number of jumps required before interstitial Mg combines with Ga or Al vacancies, we estimate the activation energy for diffusion of interstitial Mg to be around 2-2.7 eV in both materials.

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Microstructure of Mg doped GaNAs Alloys

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Successful growth of GaN_{1-x}As_x in the full composition range allowed us to explore the potential of the novel alloy system for solar device application. Monotonic change in the band gap from 3.4 to 0.8 eV with composition of GaN_{1-x}As_x alloys was observed although materials in the middle of the composition range are amorphous. It should be noted that amorphous materials could be advantageous for avoiding the formation of structural defects, especially at interfaces, that are deleterious for device application. For photovoltaic applications, demonstration of both p- and n-type doping of the alloys is needed. We report here on an investigation of the microstructures of GaN_{1-x}As_x thin films doped with Mg with As content of ~40-70% grown at temperature between 200-300°C using transmission electron microscopy.

In order to incorporate Mg in GaN_{1-x}As_x alloys we found that a higher Ga flux during the growth is required. Transmission Electron Microscopy showed that this also leads to the formation of large grains of GaAs:Mg embedded in amorphous GaN_{1-x}As_x. It is most likely that these crystalline grains are As-rich GaNAs alloys instead of pure GaAs. For two samples with ~9% Mg and approximately similar As content (66% and 62%) but grown at 200°C and 300°C, respectively, we observed the formation of large inclusions of crystalline As-rich GaNAs:Mg arranged in "chains" and embedded in the GaN_{1-x}As_x:Mg amorphous matrix. The sample grown at the lower temperature shows larger pockets of amorphous material close to the interface, just above the few monolayers of intentionally grown GaN on sapphire. The surface of this sample has GaAs:Mg grains larger than those found in the center part of this layer. TEM also showed planar defects (stacking faults and twins) present in many of these grains. The sample grown at 300C has also large elongated chains of GaNAs:Mg grains inclined to the substrate at 30-40° embedded in an amorphous matrix but the distribution of these crystalline grains is more uniform throughout the entire sample thickness. We also found that the sample grown at 300C is less conductive than the sample grown at 200°C. An increase of Mg content to about 16% with approximately the same As concentration (58%) and similar growth temperature (310C) leads to the formation of connected chains of GaANs:Mg grains and only thin areas of the amorphous material are present between these chains. This layer, however, also exhibited the highest conductivity. TEM studies on semi-insulating layers that were grown at 300C with As content of 42%, and Mg content of 10% also showed the formation of GaNAs:Mg grains distributed randomly embedded in an amorphous matrix. Correlation of the microstructures of these films with their electrical and optical properties will be discussed.

Nitrogen Rich Defects in InN

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Long believed to be non-existent in InN, we have recently explained the origin of the large quantity of excess nitrogen seen for some InN grown at low temperatures [1]. Using Arrhenius data describing the activation energy for the rate of removal of this excess nitrogen we are able to extrapolate the possible influence of nitrogen rich point defects for InN grown at higher temperatures. We conclude that although much lower quantities of these defects will be present for film growth temperatures considered optimal for InN, their presence is unlikely to be negligible even under indium rich growth conditions. We can point to recent positron annihilation spectroscopy results that support this conclusion [2]. We also present sample annealing experiments that show that InN can be improved by further ex-situ removal of excess nitrogen, with reductions in carrier concentration and an improvement in sample mobility. The implications of the presence of excess nitrogen are discussed in terms of the electrical properties of InN.

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Suppression of twin formation for the growth of InN(1013) on GaAs(110) by metalorganic vapor phase epitaxy

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In recent years, InN and its related nitrides ($\text{In}_x\text{Ga}_{1-x}\text{N}$, $\text{In}_y\text{Al}_{1-y}\text{N}$) are regarded as promising for light emitters ranging from ultraviolet (UV) to infrared (IR) region due to the superior physical properties. Currently, most of III-nitride crystals are grown on (0001) oriented sapphire. However, there is a spontaneous electrical polarization along the $\langle 0001 \rangle$ direction in wurtzite III-nitrides, which will cause the band bending and result in poor recombination probability due to the quantum confined Stark effect (QCSE) in quantum wells. A promising approach for improving the quantum efficiency of the light emitting devices is to grow nitrides along non-polar or semi-polar direction. We have reported (1013) semi-polar InN layer grown on GaAs(110) by metalorganic vapor phase epitaxy (MOVPE) [1]. Furthermore, formation of twin crystal could be suppressed by increasing the growth temperature [2]. However, decomposition of InN layer at the interface between InN and GaAs was occurred due to the relatively high growth temperature at around 600°C, which might cause the separation of the grown layer from the substrate and degradation of crystalline quality. In this study, low-temperature growth of semi-polar InN on GaAs(110) and suppression of twin crystal formation were investigated by introducing a small amount of hydrogen (H_2) in the system. Also, the mechanism of the reduction of twin crystal was proposed.

Growth of InN was performed by conventional MOVPE system. Substrate used was GaAs(110). After the thermal cleaning in H_2 ambient at 600°C followed by the substrate nitridation at 500°C, low temperature InN buffer layer (45-nm-thick) was grown at 375°C. After that, InN epitaxial layer was grown at 510°C with the partial pressures of trimethylindium (TMI) and NH_3 of 2.0×10^{-5} and 0.40 atm, respectively. During the epitaxial growth, H_2 was introduced in the range from 0 to 1% in the carrier gas.

X-ray pole figure measurements showed that the inclusion of twin crystal reduced with increasing the input partial pressure of H_2 in the carrier gas up to 0.6%, and then that increased again with increasing H_2 . The volume ratio of (1013) oriented InN to (1013) oriented InN grown with 0.6% H_2 was 0.996. The mechanism for the suppression of twin crystal can be explained by the difference in the stability of In- and N-polar InN{0001} asymmetric plane in the presence of H_2 . At low-temperature, rate-limiting reaction for decomposition of InN might be $\text{N}(\text{surface}) + 3/2\text{H}_2(\text{g}) \rightarrow \text{NH}_3(\text{g})$ according to the previous reports [3,4]. Therefore, the decomposition rate of (1013) In-polarity InN must be faster than that of (1013) N-polarity InN because of the difference in the number of back-bond to the bulk. Consequently, (1013) N-polarity InN should preferentially grow on GaAs(110) under the presence of H_2 in the carrier gas.

Surface morphology of InN layer was drastically changed by the change of input partial pressure of H_2 during the growth. Surface roughness was decreased with increasing the input partial pressure of H_2 up to 0.6%. When input partial pressure of H_2 exceeds 0.8%, formation of In droplets and degradation of surface morphology were observed. Detail mechanism will be presented in the symposium.

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3D GaN Core-Shell Structures for Solid State Lighting

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GaN nanorods and 3D columns recently attracted a lot of attention since they are expected to be an exciting new route towards light engines for solid state lighting. In contrast to a planar thin film technology, a completely 3-dimensional nano- or microrod approach gives more freedom in the device design. E.g., a core-shell design of LEDs based on 3D GaN offer a dramatically enhanced active area per wafer footprint, since the active area is scaling with height of the 3D structures. High quality core-shell devices will have a tremendous impact on LED technology.

However, there are also challenges related to a 3D device approach. Conventional planar characterization as well as processing techniques can no longer be used. In addition, the growth windows in epitaxy have to be modified in order to enhance vertical growth rates and reduce planar growth rates. Quite often, this leads to growth modes, which are far away from the ones regularly used for high efficiency planar LEDs.

This talk will give an overview on the state of the art of our 3D GaN research, particularly focusing on MOCVD growth and 3D characterization. Potential advantages and challenges of this exciting new strategy towards low cost high efficiency solid state lighting will also be discussed.

Efficiency Improvement of Nonpolar a-plane GaN Light Emitting Diodes by Controlled Integration of Silica Nano-spheres

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To improve the efficiency of GaN based light emitting diodes (LEDs), patterned substrate, surface roughening, photonic crystal and graded refractive index materials have been used. However, due to the process complexity, and hence high cost of conventional technique for efficiency improvement, the use of such process for nonpolar LED fabrication is not yet widely accepted.

In order to obtain the high efficiency nonpolar a-plane GaN LEDs by a simple process, we have reported that the controlled integration of silica nano-spheres (CIS) into the a-plane GaN epitaxial layer grown on r-plane sapphire by metal-organic chemical vapor deposition. First, we intentionally grew the 3-dimensional (3D), rough a-plane GaN buffer layer by using the anisotropic growth nature of a-plane GaN on r-plane sapphire. Then, the silica nano-spheres with diameter of 250 nm were preferentially integrated in the valley of the buffer layers by using a spin coating method. In this CIS process, the silica nano-spheres integrated in the valley act as SiO₂ mask layers in the subsequent epitaxial lateral overgrowth (ELO) process and threading dislocations originated from the GaN and sapphire interface were blocked by the integrated silica nano-spheres. Therefore, the laterally overgrown a-plane GaN layers on the silica nano-spheres were dislocation-free epitaxial layers with improved crystalline quality.

In addition to the ELO effect by the silica nano-spheres, the silica nano-spheres integrated in the GaN can improve the light extraction efficiency (LEE) of GaN-based LEDs. To confirm the effect of the silica nano-spheres on the light extraction of GaN, we investigated the diffuse reflectance of the CIS a-plane GaN epitaxial layers by collecting the light scattered at all angles by using an integrating sphere. The total reflectance of the CIS a-plane GaN was enhanced by about 30 ~ 38 % as compared to that of the conventional a-plane GaN without the CIS process in the wavelength range from 400 to 700 nm. Nanometer-scale stacked silica spheres act as internal scattering centers of the light emitted from the active layer of the LEDs. This internal light scattering changes the path of the emitted light to the top surface of LEDs. Therefore, the LEE of CIS GaN-based LEDs are increased by the enhanced probability of entering escape cone defined by the critical angle for total internal reflection.

The electroluminescence emission of CIS a-plane GaN LED was measured at drive currents ranging from 2 to 100 mA. The output power measured from the CIS a-plane GaN LED shows 130 ~150 % increase compared to that of the a-plane GaN LED without silica nano-spheres in the layer. We thus attribute the improved efficiency for the a-plane GaN LEDs to the decreased defect density in the GaN and increased extraction efficiency of the a-plane LED fabricated by CIS process.

Improved performance of blue light emitting diodes with embedded multiple layers of dielectric spheres

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We report on the improved performance of blue light emitting diodes (LEDs) with embedded layers of dielectric spheres. In this study, in order to improve the external quantum efficiency, we proposed a GaN template of inverted hexagonal cone (IHC) structure in which a larger amount of silica nanospheres can be stacked as multiple layers. The stacked silica nanospheres in the inverted hexagonal cone (SSIHC) are shown to effectively act as both excellent three-dimensional optical scatterer for the guided light and blocking masks for the propagated threading dislocations (TDs), simultaneously. It can also induce the lateral overgrowth of GaN over the dielectric layers. To closely understand the origin of improved crystal quality, cross-sectional TEM experiments for GaN on IHC and SSIHC were carried out. It is clearly seen that the SSIHC could block the TDs that are propagating along the inclined facet. Re-grown GaN above the SNs is also expected to be of higher quality since it has been grown laterally from the apices of the facets. As demonstrated in the TEM images, both the opening and the mask region of the GaN template grown on SSIHC structures have much less TDs compare to the GaN template grown on IHC structures. Another benefit of the proposed LED structure is that the light extraction efficiency is increased by scattering effect at the SSIHC due to the difference in refractive indices between GaN and silica nanospheres. As a result, the electroluminescence intensity was increased by 2.7 times at 20 mA of injection current compared with that of LEDs without silica nanospheres. This significant improvement is attributed to the combination of enhancement in both internal quantum efficiency via reduction in threading dislocations and light extraction efficiency via increased light scattering effect at the embedded stacked silica nanospheres.

Kinetics of Initial Stages of Growth of thin films of nano-structured GaN on Al₂O₃ (0001)

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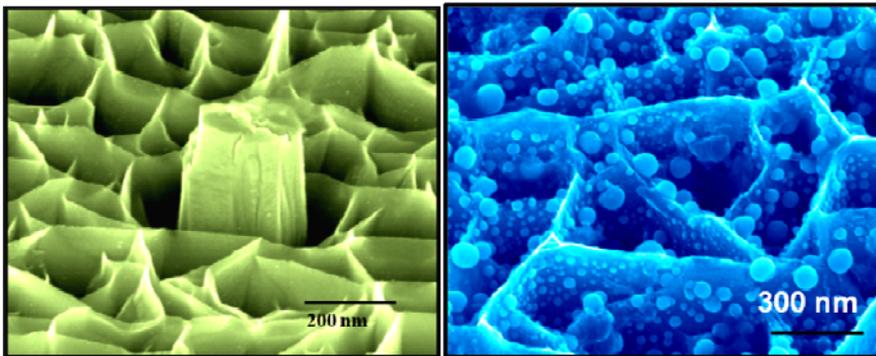
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The band gap tunability of III-nitrides, added with their novel size dependent properties in the nano-regime, offers great potential for important optoelectronic applications. The motivation behind our GaN nanostructure studies and also the current status of research will be discussed. I shall present our recent results of the MBE growth and multi-technique characterization of GaN thin films on single crystal sapphire and silicon surfaces.

Using a high-nitrogen rich condition in a PA-MBE growth, we dissuade adatoms from long range diffusion to promote super-saturation and GaN nucleation at the edge and screw dislocations, formed due to lattice mismatch, between GaN and Al₂O₃ (0001), Si(111) and (100). I shall show that this kinetically control growth results in the spontaneous formation of oriented, single crystal nanostructures such as nanowall network and nanocolumn forests, due to patterned strain relaxation pathways. The nanowall network formed around voids which are open screw dislocations, demonstrate exotic properties. Adsorption of Ag nanoparticles on the nanowall network is seen to strongly influence emission properties that manifest a coupling of the band edge emission with the Ag surface plasmons. Depending on the size and distributions of the Ag nanoparticles (see Fig), we show that we can enhance and tune Photoluminescence emission.

The nanowall network formed at the edge dislocations promote 1-D nanocolumn formations in the void between walls in a Frank type spiral growth of m-faceted GaN



(see Fig). These highly dense m-faceted nanocolumns are hexagonally shaped wurtzite, c-oriented structures and are defect controlled. We show that

depending on the kinetics of the growth, we can control the twist parameters of the Burger's vector leading to compact and defected rods, Frank-Read growth and nanotubes formation. By FFT image analysis we compute nanocolumn size, shape density and supra-structural arrangement, and used XRD and PL to their structural and optical emission properties.

Ultra High Power and RF operation of GaN/InGaN Heterojunction Bipolar Transistors on Free Standing GaN Substrates

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Power electronics based on III-nitride materials offer many advantages for operation under extreme conditions such as high voltage, current density, and temperature, as well as offering the potential for high frequency operations. In this study, we have used metalorganic chemical vapor deposition to grow *NpN*-GaN/InGaN/GaN heterojunction bipolar transistors (HBTs) on free-standing (FS) GaN and sapphire substrates that demonstrate high power and RF performance.

The layer structure consists of a GaN:Si⁺ subcollector (1 μm , $n=3\times 10^{18}\text{ cm}^{-3}$), a GaN:Si collector (0.5 μm , $n=1\times 10^{17}\text{ cm}^{-3}$), an In_xGa_{1-x}N:Si graded collector ($x=0\rightarrow 0.03$, 30 nm, $n=1\times 10^{18}\text{ cm}^{-3}$), an In_{0.03}Ga_{0.97}N:Mg base (100 nm, $p=1\times 10^{18}\text{ cm}^{-3}$), an In_xGa_{1-x}N:Si⁺ graded emitter ($x=0.03\rightarrow 0$, 30 nm, $n=1\times 10^{18}\text{ cm}^{-3}$), and a GaN:Si⁺⁺ emitter (70 nm, $n=1\times 10^{19}\text{ cm}^{-3}$). The graded layers serve to reduce strain between the interfaces, mitigate V-defect formation, and to eliminate conduction band discontinuities, particularly at the base-emitter junction.

A 20 nm low temperature GaN buffer layer for strained heteroepitaxy was included for the structures on sapphire substrate. However even with the inclusion of this buffer layer, the lattice and thermal mismatch between sapphire and GaN leads to an epitaxial structure with dislocation densities on the order of $\sim 10^8\text{ cm}^{-2}$, that inhibit the device performance, in particular via defect assisted recombination of minority carriers in the base region. But with recent improvements in technologies to produce FS-GaN substrates such as hydride vapor phase epitaxy and ammonothermal growth, homoepitaxial growth is now viable, resulting in a reduced dislocation density by two orders of magnitude to $\sim 10^6\text{ cm}^{-2}$. The improved material quality of the epitaxial structure, in terms of reduced dislocation density and V-defects, yields significantly improved device operation of the HBT. The relatively higher thermal conductivity of FS-GaN is advantageous for epitaxial growth and also acts as a built in heat-spreader for high power operation. After growth and material characterization, the layers were mesa etched via inductively coupled plasma etching, and fabricated into devices of various emitter sizes from $3\times 3\text{ }\mu\text{m}^2$ to $60\times 60\text{ }\mu\text{m}^2$. Common-emitter *I-V* family curves of a $3\times 3\text{ }\mu\text{m}^2$ structure grown on FS-GaN demonstrate a maximum collector current density (J_c) of 141 kA/cm² and a maximum d.c. power density of 3.05 MW/cm². As shown in the Gummel plot, this device can achieve a d.c. gain (β) of 82 and a maximum differential gain (h_{fe}) of ~ 120 . Due to the inherent electrical conductivity of the FS-GaN substrate, the RF characteristics of these devices are limited. However, a device with the same structure on sapphire with an emitter size of $4\times 20\text{ }\mu\text{m}^2$ was tested for high frequency operation, demonstrating a maximum oscillation frequency (f_{max}) of 1.3 GHz and a cutoff frequency (f_T) of 5.3 GHz. Development of a semi-insulating FS-GaN substrate is required for the full potential to be realized for III-N RF HBTs.

AlGa_xN layer structures for deep UV emitters on laterally overgrown AlN

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Deep UV LEDs on sapphire substrates suffer from high defect densities in AlN buffer layers. Epitaxial lateral overgrowth (ELO) of AlN on patterned c-plane AlN / sapphire is an effective approach for the reduction of the threading dislocation density down to a few 10^8 cm^{-2} . The application of such ELO-AlN templates for the deposition of AlGa_xN layers with high Al mole fractions and its influence on their properties will be shown. The epitaxially grown AlN layers exhibit an excellent transparency down to 210 nm enabling bottom emitting structures in the deep UV.

The (Al,Ga)N layers were deposited by MOVPE in a multi-wafer Aix2400G3HT planetary reactor with 11x 2 inch satellites using the standard precursors TMAI, TMGa, ammonia and hydrogen as carrier gas.

The crystalline perfection of AlGa_xN layers with Al-contents higher than $x > 0.4$ on ELO-AlN was investigated by high resolution X-ray diffraction (HRXRD), photoluminescence (PL), cathodoluminescence (CL) and resistivity mapping (MRES). The critical layer thickness of AlGa_xN on ELO-AlN is drastically increased compared to AlGa_xN deposited on planar AlN/sapphire templates. Cathodoluminescence mapping reveals compositional variations caused by different Ga incorporation during faceted growth over steps from step bunching of the ELO-AlN. With increasing Al-content the homogeneity is improved.

Additionally, the n-type conductivity of silicon doped AlGa_xN layers with Al contents up to $x = 0.82$ was investigated by resistivity measurements. While layers on planar AlN buffer are highly resistive, ELO allows for efficient Si-doping. For 1.8 μm thick n-AlGa_xN the sheet resistivity is around 260 Ohm/sq. for $x = 0.77$ and increases to 750 Ohm/sq for the same IV/III ratio in the gas phase if the Al content increases to 0.82.

Finally, the influence of the ELO-AlN template on the internal quantum efficiency (IQE) of AlGa_xN/AlN multi-quantum-well structures emitting between 250 nm and 224 nm was studied by temperature dependent PL measurements. For emission around 230 nm the IQE improves by a factor of about two for MQWs on defect reduced ELO AlN. The data show the usefulness of defect reduced ELO-AlN templates as quasi-substrates for AlGa_xN layer structures with high Al-content that can be employed in LEDs emitting in the deep UV.

CHVPE growth of AlGaIn-based UV LEDs

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Ultraviolet (UV) light-emitting diodes (LEDs) have gained considerable interest due to a number of applications, e.g., water and air purification, UV curing, identification of chemical and biological agents. In this paper we report the results of manufacture of our 360-365 nm AlGaIn-based UV LEDs.

The external quantum efficiency (EQE) of AlGaIn-based UV LEDs with emission wavelength shorter than 365 nm is at least one order of magnitude below the best devices in the near UV and violet wavelength range. Another problem of UV LEDs is high cost of manufacturing process. Chloride-hydride vapor-phase epitaxy (CHVPE) provides much lower growth costs than metalorganic chemical vapor deposition (MOCVD) and molecular-beam epitaxy (MBE). Additional advantages potentially leading to high internal quantum efficiency (IQE) in CHVPE-grown UV LED structures are due to the low impurity contamination. CHVPE is a “carbon-free” technology and HCl used in the CHVPE process provides an impurity-cleaning effect during CHVPE growth.

In conventional LEDs the InGaIn active region cannot be made thick enough because of strain relaxation via generation of misfit dislocations. In indium-free LEDs much thicker active regions can be utilized, which mitigates technological requirements on interface quality, as compared to quantum-well LED heterostructures. We use a thick indium-free layer as an active region, which is a reasonable choice for high-current operations because in the structures with multiple quantum wells (MQW) the problem of inhomogeneous injection of electrons and holes to different wells has not been resolved yet and the droop of the IQE is observed even at low current densities.

Two-inch-diameter sapphire (0001) substrates were used for the growth of the UV LED structures. The structures were grown employing a CHVPE growth machine equipped with an atmospheric pressure quartz hot-wall reactor and a resistively heated furnace. Samples were grown at about 1050° C using Ga and Al metals as source materials and HCl and NH₃ as gaseous reagents. Ar was used as a carrier gas. Doping was done by introducing Mg (p-type) or Si (n-type) impurities in the growth zone of the machine. From bottom to top, each LED consists of an undoped grading AlGaIn structure, n-AlGaIn hole-blocking layer, n-AlGaIn light-emitting layer, p-AlGaIn electron-blocking layer and p-GaN top layer. The processing and packaging were done using outside service.

The grown structures were characterized by X-ray diffraction (XRD), secondary ion mass spectrometry (SIMS), electroluminescence (EL), atomic force microscopy (AFM). Device structures emit at peak wavelengths of 360-365 nm. For the p-type AlGaIn layers Mg atomic concentrations are in the range of 10¹⁹-10²⁰ cm⁻³. Threading dislocation density (TDD) in epitaxial layers is (5-7)·10⁸ cm⁻². Diced individual LEDs with dimensions of 310 μm × 310 μm operate at 20 mA current and reveal 3 mW optical power.

Development of Micro-Plasma Excited Deep Ultraviolet Light Emitting Device (MIPE-DUV Light Emitter) using AlGa_N grown by Metal Organic Chemical Vapor Phase Epitaxy

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Deep UltraViolet (hereafter DUV) light emitting devices are very important and useful for real application for sterilization of water, tools used in hospital, ozone sensing, therapy of skin allergy, new production of chemical materials, and so on. So far, for developing DUV light emitting devices, AlGa_N light emitting diode, and electron beam excited light emitter based AlGa_N have been proposed and realized and partly commercialized. However, the energy efficiency of these devices is less than 3 to 4% at maximum and is much less than that of commercially available white light LED. In addition to this low efficiency, enlargement of these devices are quite difficult. Otherwise, plasma display flat panel (hereafter PDP) has large light emitting area and total emitting light power is very strong. In this presentation we report a trial of **micro plasma excited DUV light emitter (MIPE-DUV Light Emitter)** using AlGa_N quantum well grown by metal organic chemical vapor phase epitaxy (MOCVD) for the first time.

Devices consist of three parts; electrodes, plasma sustain part and DUV light emitting part. Electrodes are fabricated using Au/Cr electrodes covered by dielectric material and MgO. MgO is essentially important to realize easy plasma formation due to the high secondary electron emission yield of MgO. Plasma is sustained using some time sequence of applied voltage and for efficient plasma formation this sequence is very important. Gas used is a mixture of Xe and Ne. The concentration of Xe is 10 to 20%. The light emission from Xe and Ne mixture of around 172nm excites directly AlGa_N quantum well surface and electron from plasma was accelerated using a time sequence of applied voltage and this electron also excites AlGa_N quantum well. Gas pressure used is around 1E2 to 5E4 depending on the cell structure.

We clearly observed emission from the quantum well. When we used Xe and Ne mixture, the emission from quantum well and barrier layer were observed, but we used only Ne we could observed the emission from the quantum well. In case of the mixture of Xe and Ne the light of 172nm from the plasma of the mixture is absorbed at the quantum barrier surface, which is the top surface of the AlGa_N light emitting material, and quickly recombined at the surface of the barrier. The emission wavelength of the barrier is around 265nm which is almost similar to the main peak of the emission of photoluminescence in our sample used. In case of Ne only plasma, no light excitation exists and the electron beam excitation is main process. In this case the electron has high momentum and easily diffuses into the quantum well and emits the light from the quantum well at about 296nm which is the same as the wavelength of the light emission from quantum well observed in photoluminescence measurement as the second peak.

The intensity and the efficiency has not yet been determined at this moment, but in principle the size enlargement of our MIPE-DUV light emitter has no limitation. So, our MIPE-DUV light emitter may have large area and strong total emission.

Fabrication of 2'' freestanding GaN substrates by HVPE using low temperature buffer layer on sapphire

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The III-nitrides are getting considerable attention because of their applications, e.g. for high-brightness blue and green light-emitting diodes and room-temperature continuous-wave blue laser diodes. So far the III-nitride devices on the market are grown on *foreign* substrates, like sapphire, SiC or Si. This leads to very stressed GaN layers with high density of defects due to the large lattice mismatch and difference in thermal expansion coefficient. Thus, freestanding GaN substrates of large area and with low density of defects are highly desirable for the next generation of III-nitride based devices. In order to fabricate freestanding GaN substrates with low stress and defect density, the HVPE growth technique have so far been most successful.

In this work, we have studied growth and self-separation of bulk GaN on *c*-oriented Al₂O₃ using low temperature GaN buffer layers (LT-GaN). The growth was done in a vertical HVPE reactor at atmospheric pressure where the carrier gases and precursors are delivered from the bottom. GaCl, NH₃ and N₂ or a mixture of N₂ and H₂ is used as gallium source, nitrogen source and carrier gas, respectively, in the experiments. The GaCl is synthesized in situ by flowing HCl over a quartz boat containing liquid gallium held at 700-750°C. In order to estimate the partial pressure of process gases at the substrate surface and assure laminar flow conditions, numerical simulations were done. For additional details about reactor and simulations, see ref [1].

A number of GaN films with thickness ranging between 20 μm and 3 mm were grown onto 2-inch sapphire substrates. The LT-GaN buffer layers were grown at 600 °C using a V/III ratio in the range of 17-70 at the substrate surface as determined by simulation. The growth time of the LT-GaN buffer was varied between 90 sec and 9 min which correspond to a thickness variation about 70 nm to 350 nm, respectively. The bulk GaN layers were grown at 950-1000 °C with a growth rate about 200 μm/hr. To study the structural and optical properties of the buffers and the GaN layers, low temperature photoluminescence, X-ray diffraction (XRD), cathodoluminescence, optical microscopy and reflectance spectroscopy have been used. We have obtained the highest structural quality of the bulk GaN (XRD ω peak with a full width at half maximum of ~450 arcsec for a 20 μm thick GaN layer) using a buffer growth time about 3-6 min. By studying the in-plane and out-of plane strain versus the LT buffer thickness of the layers, we observe that the strain in the layers is minimized using a LT buffer thickness about 100 nm-250 nm which also is the range where we obtain the highest structural quality of the GaN. By using an optimized LT-GaN buffer layer, thick and crack free freestanding 2'' GaN substrates were manufactured by spontaneous self-separation.

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The effect of surface state of the seed substrate on the growth of GaN single crystal by Na flux method

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High quality GaN crystals with low defect density are desirable for the fabrication of high-performance optical and electrical devices. We have studied the growth of GaN crystals by Na flux method, which has dislocation reduction mechanisms during the liquid phase epitaxial (LPE) growth [1]. In this study, we were focused on surface state of seed substrates before LPE, and examined appropriate states which actualize high quality GaN crystals.

In this study, c-plane seed substrates were treated in three different ways: (a) mechanical polishing (MP), (b) chemical mechanical polishing after mechanical polishing (CMP) and (c) wet etching using pyrophosphoric acid after mechanical polishing (WE). A seed substrate was placed in an aluminum oxide crucible, and then, metal-Ga, metal-Na and carbon grains were set in the crucible in an Ar-filled glove-box. The crucible including the starting material was transferred into a stainless-steel tube. The temperature and nitrogen pressure of the tube were maintained at 870 °C and 4.0 MPa, respectively, during the growth period for 96 h. The crystallinities of seed substrates treated by each method and GaN crystals grown on each substrate were evaluated by the full width at half maximum (FWHM) of X-ray rocking curves (XRC). Dislocation densities of grown GaN crystals were investigated by panchromatic cathode luminescence (CL) measurement.

Crystallinity of seed substrates treated with CMP and WE were remarkably improved comparing with that of seed substrates treated with MP only. The dislocation density of the seed substrate was $3.7 \times 10^7 \text{ cm}^{-2}$, and that of the GaN crystals grown on the seed substrates treated with MP, CMP and WE were 1.7×10^6 , 2.2×10^5 and $4.6 \times 10^4 \text{ cm}^{-2}$, respectively. WE treatment was the best of the three methods to achieve high crystallinity and low dislocation density. The results possibly indicate that damaged layers caused by MP treatment were removed by the two treatment methods, leading to the improvement of crystallinity of the substrate surface and the LPE GaN crystals. The difference of dislocation density can be explained as below. Dislocation density increases at interface between a LPE GaN layer and a seed substrate when the state of the seed substrate is bad condition (ex. MP). However, if the state of substrate is improved, such the new dislocations do not occur, thus the dislocation reduction mechanism of Na flux method effectively lowers dislocations of LPE GaN crystals. Next, we grew GaN crystal on the GaN crystal grown on the seed substrate treated with WE. Before the second growth, the surface of the GaN crystal was treated with WE in the same way as the seed substrate. The final dislocation density of the GaN crystal was $3.9 \times 10^3 \text{ cm}^{-2}$. This result indicates that the dislocation density of GaN crystals probably decreases by repeated growth.

We conclude that the superior surface state treated by pyrophosphoric acid is effective and available for the fabrication of GaN substrates suitable for the growth. This technique enables us to grow GaN crystals repeatedly by Na flux method, attaining GaN single crystals with lower dislocation densities.

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Coalescence of GaN nanorods for high quality templates

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The lack of commercially available GaN substrates has prompted the use of sapphire, silicon and SiC as platforms for the growth of nitride semiconductors. This leads to large lattice and thermal mismatches which, in turn, result in a high density of threading dislocations, a large accumulated strain, and a significant bow of the wafer. These phenomena create non-radiative recombination centers, limit the overall devices lifetimes, and prevent up-scaling to industrial production.

In order to get round these limitations, in this work a novel epitaxial growth procedure based on GaN nanorod coalescence is proposed and explored. The nanorods, which act as seeds for the epitaxial overgrowth, represent a very efficient means of filtering threading dislocations and, due to their large free surface area, allow complete strain relaxation, even on Si substrates.

To systematically study the properties of the nanorods and, in particular, to avoid large size dispersions typical of bottom-up nanorods, a top-down approach (homogeneous over 4 inches sapphire and silicon substrates) has been employed to fabricate them.[1,2] The coalescence of these nanorod templates is done on a MOVPE reactor using a two-step process. Indeed, after the formation of a pyramid on top of each nanorod (step1), the growth conditions are changed to promote bi-dimensional growth (step2).

As a starting point, the structural properties of the GaN nanorods have been determined using Scanning Electron Microscopy, Photoluminescence, and high-resolution X-ray diffraction. Then, the influence of the nanorods height and pitch on the final template quality has been assessed. Similarly, the switching conditions between the step 1 and 2 have been optimized and correlated with the final template quality. Finally, the evolutions of the threading dislocation density and of the strain state have been determined as a function of final GaN layer thickness (from 500nm to 5 μ m). For 1-2 μ m thick templates, dislocation densities as low as 3x10⁸ cm⁻² and 5x10⁸ cm⁻² have been obtained on Sapphire and Silicon, respectively. These high-quality GaN templates can become the platform for high-efficiency LEDs.

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Stress distribution in 12 μ m thick continuous GaN grown on patterned Si substrate

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Group III-nitride semiconductors are of great interest for the applications in high power, high frequency electronic devices and in related technologies such as light emitting diode (LED), laser diodes [1,2]. The growth of GaN on silicon is favorable because of the low cost, large size and good thermal conductivity of the silicon. However, the large lattice mismatch (17%) between GaN and Si leads to high dislocation density and the thermal expansion co-efficient mismatch (~56%) is responsible for the crack formation during cooling down if the layer is more than a critical thickness [3,4]. One of the approaches to grow crack free GaN is to pattern the Si substrate [5,6,7]. Here the effects of the patterns on the thick GaN film and the stress distribution of the crack free thick GaN mesas are investigated by the micro-photoluminescence (μ -PL) at low temperature and the micro-Raman spectroscopy.

In the experiment, Si (110) and Si(111) substrates are patterned using UV lithography followed by chemical etching (with KOH) or reactive ion etching (with SF₆) and different shapes such as triangle, square, lozenge and hexagon and different sizes (50 μ m up to 400 μ m of length) of the patterns are obtained. Up to 12 μ m thick crack free continuous GaN layers are grown by metal organic chemical vapor deposition (MOCVD). Scanning along the long diagonal axis from the corner towards the center of the 400 \times 400 μ m² lozenge shaped mesa is performed by μ -PL measurement at low temperature (4K) and this gives a red shift of D⁰X_A (donor bound exciton) energy which indicates more relaxed GaN at the corner while the center is under tensile stress. The same trend of stress distribution is observed from the stress measurement by the micro-Raman spectroscopy. It is also found that the stress varies with the size, shape and the trench height of the pattern with variations as high as 0.5 GPa.

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The coalescence growth of GaN crystals on point seed crystals using Na flux method

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GaN has attracted attentions because of their application in optical and electronic devices with low power consumption. Fabrication of large high-quality GaN single crystal substrates, however, is not realized. We have grown GaN crystals using Na flux method, by which the dislocation density decreases 10^8 cm^{-2} in a seed to 10^4 cm^{-2} in liquid phase epitaxial growth (LPE) [1]. Recently, extremely low-dislocation GaN crystals were grown using “GaN point seed”, which allows the elimination of dislocations propagating from a seed layer by necking technique. In this work, we developed a new process, the coalescence growth of isolated point seeds, in order to fabricate large diameter GaN wafers with extremely low dislocation density.

Point seed crystals were established by mounting a sapphire plate with small holes freely on a c-GaN substrate. The full width at half maximum (FWHM) of X-ray rocking curve (XRC) of the seed was 198 arcsec. Two point seeds 1.2 mm in diameter were arranged in two patterns; these were arranged along the m-direction (m-direction coalescence) and the a-direction (a-direction coalescence) of the GaN with a pitch of 0.5 mm, respectively. The point seeds, Ga, Na and Carbon were placed in a crucible. Carbon was added to suppress heterogeneous nucleation. The growth proceeded with nitrogen dissolution into a Ga-Na melt at 870 °C under N₂ pressure of 3.6 MPa for 96 - 200 h. XRC measurements were performed to investigate crystallinity. Dislocation density was investigated by panchromatic cathodoluminescence measurements at room temperature.

GaN crystals grown on the two point seeds coalesced in the both arrangement. In the arrangement of the m-direction coalescence, voids were incorporated in the coalescence boundary. By contrast, in the arrangement of the a-direction coalescence, GaN crystals coalesced without voids. The full width at half maximum (FWHM) of XRC (0002) of the GaN crystal coalesced along a-direction was about 55 arcsec, which was much narrower than that of the seed (198 arcsec), at any region including the coalescence boundary. In panchromatic CL images, no coalesce lines were apparently observed. The dislocation density was less than $6.9 \times 10^3 \text{ cm}^{-2}$ in large area of the coalesced crystal.

We also attempted to grow GaN crystals from three point seeds with a triangle lattice-line. Each point seed was arranged so that a-direction of a point seed corresponded to that of the neighboring point seeds. As the result, the three GaN crystals coalesced without voids in coalescence boundaries.

We concluded that the coalescence growth of periodically-arranged point seeds is an effective way for fabricating large and high-quality GaN crystals.

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Solid-source solution growth (3SG) method for bulk AlN growth

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AlGa_N has attracted much attention for applications in low-energy-loss power devices and deep UV light-emitting-diodes (DUV-LEDs). In the AlGa_N films grown on AlN/sapphire or SiC substrate, however, dense threading dislocations, which influences on the device performances, were generated at the hetero interface. Therefore, it is indispensable to develop a high-crystalline-quality bulk AlN in order to achieve homoepitaxial growth and suppress the dislocation generation.

Recently, researchers have examined the growth of bulk AlN using a solution growth method which enables a low-temperature growth of the semiconductor. For example, Isobe et al. [1] grew a 1.8 μ m-thick AlN on AlN/sapphire at 900°C using Ca-Sn-Al flux. Then, Adachi et al. [2] grew a 1 μ m-thick AlN on nitrided sapphire substrate at 1300°C using Ga-Al binary solution. These growth methods for bulk AlN, however, used N₂ gas as a nitrogen source. In this case, the solubility limit of nitrogen at the vapor-liquid interface limits the nitrogen composition in the solution. This limit reduces the range of available supersaturations, which limits the growth rate. In the present work, we investigated the feasibility of a solid-source solution growth (3SG) method for the growth of bulk AlN. In our 3SG method, Li₃N instead of N₂ gas was used as a nitrogen source. By this method, we can control the nitrogen composition in the Al-Li₃N solution, and a fast growth of bulk AlN under moderate conditions would be possible.

The bulk AlN was grown by the 3SG method using powdered Al and Li₃N as source materials. An AlN seed crystal was placed together with source materials in a molybdenum crucible. The seed crystal was a self-nucleated, faceted AlN crystal grown by physical vapor transport [3], and the composition of the source materials was Al:Li₃N = 4:1. The crucible was then heated up to 1250°C in an atmospheric pressure of nitrogen, then slowly cooled over the course of 10 h to 1200°C. During this slow cooling process, AlN grew on the seed crystal.

Using the 3SG method, a thick AlN layer was grown on an aggregated of self-nucleated columnar AlN crystals. The growth rate, GR, was so high that the thickness reaches ~5 μ m in 10 h, i.e., GR = ~0.5 μ m/h. The GR (=~0.5mm/h) is faster than those denoted in other reports [1,2] (GR = 0.02 – 0.2 μ m/h). The TEM analysis showed that the AlN layer epitaxially grew on the (1-100) side-wall of the columnar seed crystal and contained threading dislocations (TDs) propagating along [1-100]. The density of TDs was as large as ~10⁹ cm⁻² in the lower region of AlN layer but decreased down to be ~10⁸ cm⁻² in the upper region. The reduction in density was confirmed to be due to the formation of dislocation dipoles or half-loops. These results suggest that the 3SG method can grow relatively high-quality bulk AlN under moderate conditions (temperature: $T = 1250 - 1200^\circ\text{C}$, N₂ pressure: $p = 1\text{atm}$).

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Growth of Bulk AlN using Alumina Reduction Method

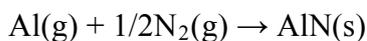
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AlN is an attractive substrate material for ultraviolet laser diodes (UV-LDs) and light emitting diodes (UV-LEDs), because it has some outstanding features such as high transparency of UV light, high thermal conductivity and good lattice matching to AlGaIn devices. However, large bulk AlN crystal has not been commercially available yet with reasonable cost. A sublimation method [1-3] is one of the promising methods to grow bulk AlN crystal. The sublimation method requires a high growth temperature ranging from 2100 to 2250 °C to obtain higher growth rates. To decrease growth temperature, Hironaka et al attempted the AlN growth using carbothermic reduction of alumina in a nitrogen gas atmosphere [4]. In their method, alumina powder was carbothermically reduced to form Al₂O gas at 1950 °C, and AlN was grown by the reaction between Al₂O and nitrogen gases at a lower temperature area. Thus, they demonstrated the possibility for a low-temperature bulk growth process using alumina reduction.

We have continued studying the alumina reduction method. In the present study, a sintered alumina plate (25 mm × 50 mm × 1 mm) was heated in a graphite chamber, and nitrogen gas was blown at a flow rate of 0.5 L/min on the alumina at 1950 °C. After the 30-h-growth operation, AlN crystal grew around the alumina plate. The nucleation of AlN crystal occurred on the edge of the alumina plate, and AlN grew toward the center of the plate as can be seen through optical and scanning electron microscope observation. A piece of AlN crystal obtained at 1950 °C was characterized, and its full width at half maximum (FWHM) value of AlN(0002) X-ray rocking curve (XRC) was 64 arcsec.

The growth mechanism is considered as follows. Alumina thermally decomposed, and the equilibrium partial pressures of Al(g) and Al₂O(g) are estimated as 6.0×10^{-8} and 1.1×10^{-8} bar, respectively, at 1950 °C assuming stoichiometric decomposition. The equilibrium partial pressure of carbon is estimated as 2.3×10^{-9} bar at 1950 °C, which may contribute to the alumina reduction. Thus, the alumina decomposes at the process temperature, and can be enhanced by lowering oxygen partial pressure in the carbon-saturated nitrogen gas. The Al(g) reacts with the nitrogen gas forming initial nucleation of AlN by the following reaction.



After nucleation of AlN, small AlN crystals coalesce and grow toward the center of the alumina plate. The growth direction depends on the nitrogen gas flow pattern in the chamber, which should be optimized to obtain large crystal. At the symposium, more details on the process and crystal characterization will be presented.

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Molybdenum as local growth inhibitor in ammonia based epitaxy processes

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Growth of thick gallium-nitride (GaN) layers in horizontal hydride vapor phase epitaxy (HVPE) typically suffers from inhomogeneous growth and severe edge effects. Especially edge effects can decrease the usable area of 2"-wafers with thick top layers. Typically, GaN grows in very bad shape or even polycrystalline in a ring next to the wafer edge. Moreover, the growth is not limited to the wafer topside but may nucleate also on the wafers side wall.

Our investigations focus on the growth of thick GaN layers that separate from the sapphire templates during the process cool-down because of the difference in expansion coefficients of sapphire and GaN. For this to happen we have developed a weak interlayer near the sapphire-GaN interface based on a structured dielectric mask. Working well in general this method typically fails at the wafer edge because of the mentioned GaN growth on the wafer sidewall. This additional material is clamping the top layer of GaN to the sapphire below, counteracting the effect of the weakened interlayer.

This unwanted growth may be prevented by masking the wafer sidewalls adequately. However, experiments using traditional mask materials like SiO_x or Si_xN_y have failed for thick layers. From earlier experiments we know that molybdenum is one of the rare materials withstanding the harsh process conditions of the HVPE. Therefore we carried out our first experiments with this material as a mask.

Indeed, positioning a Mo ring around the wafer already resulted in no growth taking place in the vicinity of the metal ring along the wafer edge for samples with an overall layer thickness of at least 300μm. As expected this method also helped improving the self-separation process greatly. Additionally, the formation of polycrystalline GaN near the edge was significantly reduced.

From a chemical point of view, we suppose that nitrogen provided by ammonia is locally removed from the process by turning the molybdenum to molybdenum-nitride. When using thin films of sputtered molybdenum we have observed that the metal is consumed in the process. No visible trace of the 150nm thick layer of sputtered molybdenum could be found after a three hour growth run at standard c-plane growth conditions. A SEM cross section picture of the respective 300μm thick sample shows a thin polycrystalline layer of GaN in place of the molybdenum.

Although the molybdenum-nitride seems too dissolve at process conditions we have found no evidence of the molybdenum being incorporated into resulting layers. In earlier experiments this has been proven by SIMS measurements of GaN layers grown with molybdenum being in the reactor.

Gallium nitride crystal growth in situ monitoring techniques using bulk material optical properties

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Most non-contact insitu monitoring techniques depend on the surface properties of both the underlying substrate and the crystal being grown in order to estimate real time surface temperatures and infer other growth properties. In these methods, the emissivity of the substrate is most often assumed to be known and serves as reference for the changes in surface optical properties during growth. However depending on the surface preparation of these substrates this assumption may not be valid, especially if a high level of precision is required of the measurement. Furthermore any change in the optical path of the light being used to detect these properties, as well as changes such as window transmission due to material condensation during growth, may result in unpredictable errors.

A technique based on the bulk band-edge transmission of gallium nitride which can be used both to calibrate pyrometers, as well as to precisely monitor changes in absolute temperature during the growth of gallium nitride crystal is described. At high acquisition rates, it is a candidate method for precise insitu spatially resolved surface temperature monitoring. Moreover as the measurement principle is not based on radiative heat emission, it may have applications at low temperatures difficult to attain using pyrometric methods.

The instrument uses single or multiple wavelengths of laser light aimed at the surface of the wafer during growth while non-specular reflection is monitored. This ensures that most of the signal reaching the instrument's detector is in fact light which has been diffusely reflected from the back of the substrate, and therefore characteristic of the transmission from the bulk crystal. The wavelengths are chosen such that they are at or near the band-edge of gallium nitride at the temperature where monitoring is sought. Since many gallium nitride based structures are grown following several microns of non-intentionally doped material, the transmission will typically be that of undoped gallium nitride and not depend much on the details of the following (optically thin) structure. As the temperature of the substrate sweeps through the temperature at which the band-edge matches the monitoring wavelength, a variation of the monitored signal can inversely be attributed to the wafer reaching that temperature.

As a calibration tool, this can then be used to adjust other relative temperature monitoring instruments, such as pyrometers. Successfully implemented, no interruption of the growth cycle would be necessary for calibration as the growth of the gallium nitride crystal would be used to self-calibrate the instruments.

Finally the use of two monitoring wavelengths further enables distinction from surface effects which could in principle also affect the monitored signal. If one wavelength is set near the band-edge while the other far from enough from it, changes in the first signal relative to the second can be assumed to be due to temperature changes. When both change in a similar fashion, the change can be attributed to non-temperature effects such surface roughness.

Ammonothermal crystal growth of GaN using an NH₄Br mineralizer

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Gallium nitride (GaN) is a wide band gap semiconductor with unique material and electronic properties. It has wide applications as the key material for manufacturing next generation high-power and high-frequency devices. To meet the great demand for high-quality GaN crystals, a variety of methods, such as hydride vapour phase epitaxy (HVPE), Na-flux, N₂ high pressure, and ammonothermal method, have been proposed for the crystal growth of GaN. Among those methods, the ammonothermal one has gained increased recognition as the most promising method to realize mass production of GaN crystals due to its advantages including scalability, crystal quality and cost effectiveness, even though it is the most recent proposed route.

Mineralizer plays an important role in the ammonothermal growth of GaN since it determines the chemistry of crystal growth. Both basic and acidic mineralizers have found applications in ammonothermal crystal growth of GaN. In the past few years our studies are focused on the using of acidic mineralizers, including NH₄Cl, NH₄Br and NH₄I. Examples of ammonothermal growth of GaN using NH₄Cl and NH₄I as mineralizer and characterization of as-grown GaN crystals have been reported. However, examples of ammonothermal crystal growth of GaN in the presence of NH₄Br are still absent from the literature. In this communication, the NH₄Br-mineralized ammonothermal crystal growth of GaN was reported.

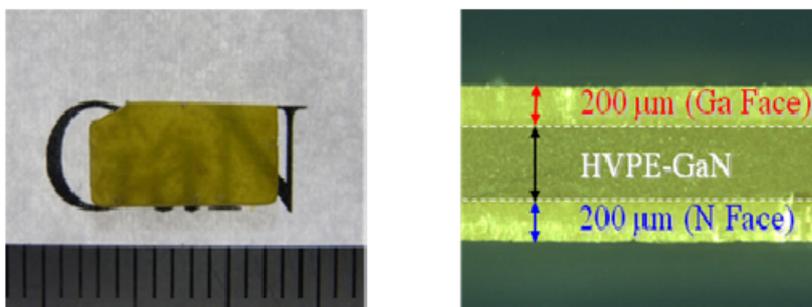


Figure 1. Images of as-grown GaN crystals using NH₄Br as mineralizer

N-type doping of HVPE-grown GaN boules using dichlorosilane

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GaN boules grown by Hydride Vapor Phase Epitaxy (HVPE) can serve for cutting of substrates with strongly reduced dislocation densities and different crystal orientations. N-type conductivity is desired in order to take full benefit of such GaN substrates for the fabrication of opto-electronic devices like laser diodes. The dopant silicon is commonly used in the growth of epitaxial device structures grown by MOVPE or MBE although it is observed to go along with an increase of tensile strain that may eventually result in layer cracking along {1-100} planes at reduced crack-free layer thicknesses compared to undoped layers. But growth without crack formation is essential for successful fabrication of GaN boules by HVPE. The use of other dopant elements like oxygen or germanium has been proposed but they were found to come along with either a higher activation energy or an inferior incorporation efficiency into GaN layers compared to silicon. The aim of this study was to consider the compatibility of the dopant silicon for growth of GaN crystals of several mm in thickness.

HVPE growth was performed in a commercial vertical HVPE reactor (Aixtron) on MOVPE-grown GaN layers of about 4 μm thickness on (0001) sapphire substrates. A series of single GaN layers of about 1 mm in thickness using various flows of a dichlorosilane/hydrogen mixture was grown on a non-intentionally doped GaN buffer of about 200 μm thickness. The growth rate of these n-type GaN layers was about 400 $\mu\text{m}/\text{h}$, the growth temperature 1010°C and the total reactor pressure 200 hPa. The input V/III ratio was about 12. Complete spontaneous separation of the layers from the sapphire substrates during the cool-down process resulted in large transparent sample pieces of about 1.2 mm thickness with smooth surfaces. Samples of 5 x 5 to 10 x 10 mm^2 were sawn and more than 200 μm were polished off from the backside. Hall-effect measurements revealed n-type conductivity with high mobilities and carrier densities of 400 cm^2/Vs at $8.7 \times 10^{17} \text{ cm}^{-3}$ to 200 cm^2/Vs at $1.5 \times 10^{19} \text{ cm}^{-3}$. The oxygen background was below the SIMS detection limit of about 10^{16} cm^{-3} and the electrical activation of the incorporated Si was found to range between 80 and 90%. FWHM of rocking curves for the symmetric 002 reflection related to screw dislocations was found to increase from 80'' to 160'' from the lowest to the highest doping level but the skew-symmetric 302 reflection related to mixed and edge dislocations had constant FWHM of about 90''. The lattice constants match well to published data for thick and freestanding state-of-the-art non-doped GaN layers and no tensile strain was indicated. These findings show that silicon is well suited for n-doping of GaN boules. The growth was prolonged with moderate doping of about $1 \times 10^{18} \text{ cm}^{-3}$ up to a thickness of about 7 mm resulting in coverage by V-pits quite similar to surfaces of comparable non-intentionally doped GaN crystals. Different origins of such V-pits have been identified using m-plane cross-sections of GaN crystals. Results of further growth optimization to minimize surface defects and to increase the useable thickness of the Si-doped GaN crystals are presented.

Free-Standing GaN Wafer by HVPE with a Pit-Induced Buffer layer

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High-quality GaN substrates are most indispensable for high performance LEDs and LDs. GaN wafers have been produced by many kinds of separation methods from sapphire substrate using hydride vapor phase epitaxy (HVPE) [1-2]. HVPE is a very attractive growth method owing to its high-volume and low-cost production [3]. However, free-standing GaN (FS-GaN) substrates are still expensive due to the difficulty of manufacturing process. The separation techniques require a complex process such as deposition of metal material, mask patterning and growth of GaN template by metal organic vapor deposition, prior to the growth of thick GaN film by HVPE. Recently, one-stop HVPE process for the growth of FS-GaN thick layers using evaporable buffer layer (EBL) has been demonstrated [2], though two-inch dia crack-free GaN thick layers has not been reported yet. Additionally, this technique requires a critical control of the density of GaN dots on nitrated sapphire to enable lift-off of the sapphire substrate. Because of those factors, a cost competitive process for FS-GaN has not been established yet. This paper will demonstrate a one-stop HVPE process for the fabrication of FS-GaN using pit-induced LT-GaN buffer.

GaN film was grown by conventional HVPE on 2-inch dia *c*-sapphire. NH₃ was used as the N source. Ga metal was supplied as GaCl, a product of the reaction with HCl. Ga metal source was maintained at 800°C during growth process.

The process began with nitridation of *c*-sapphire substrate at 1150°C, while N₂ was used as carrier gas. The substrate temperatures were cooled down to grow low temperature GaN (LT-GaN) at 500°C. Next, the substrate temperature was increased to 700°C to grow a pit-induced buffer layer. Finally, high temperature GaN (HT-GaN) was grown after changing the carrier gas to H₂.

Scanning electron microscope (SEM) observation on the surface of a 5- μ m-thick HT-GaN film shows that intentionally induced pits are observed with a density of 5×10^9 cm⁻². The HT-GaN layers cover the pits as growth proceeds. A 1.7-mm-thick GaN films were separated from sapphire substrates during cooling process. As a result, FS-GaN wafer is obtained successfully by one stop. We stress that the self-separation of GaN thick layers has been reproducibly achieved. In conclusion, we have succeeded in the fabrication of FS 2-inch dia GaN thick wafers by one-stop HVPE using pit-induced LT GaN buffer, which will lead to a low-cost fabrication of FS-GaN wafers.

The authors would like to thank to Prof. K. Fujii of Tokyo University for fruitful discussion.

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GaN growth modes and corresponding macrodefects

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Thick (millimeter range) GaN layers were grown by HVPE method. Two growth modes have been observed. The dependence of the growth mode on growth parameters is studied and macrodefects corresponding to different growth modes are described:

i) At high growth temperatures or low growth rate GaN layers with smooth surface and high tensile growth stress were obtained. Tensile stress leads to crack formation in GaN layer during growth.

ii) High growth rate at lower temperatures leads to significantly reduced growth stress and rough surface morphology. In this mode crack formation can be avoided however V-shaped defects (pits) develop on the surface of film. Influence of growth parameters on the pit shape evolution was investigated. Origins of V-defects formation are discussed.

Combination of growth modes allowed to obtain crack-free layers with smooth surface and reduced pit density.

Powder synthesis of GaN using Ga metal as a starting material

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High-purity powder is in great demand because of its unique physical properties such as wide band gap and excellent transport capacity. Using Ga as a starting material for powder synthesis and ammonothermal crystal growth can reduce the concentration of impurities. However, Ga is not a preferred choice because direct reaction of metallic Ga with NH_3 in a simple manner could not be accomplished even at 1000 °C owing to the formation of a thin layer of GaN crust on the surface of molten Ga, which prevents the diffusion of NH_3 into the unreacted Ga.

In this study, synthesis of GaN powder using metallic Ga as a raw material in the presence of NH_4X (X: F, Cl, Br and I) was investigated. It was found that ammonium halides can catalyze the ammonolysis of Ga to GaN in supercritical NH_3 . NH_4I was the most efficient catalyst among the ammonium halides, and the conversion of Ga depended significantly on the amount of NH_4I and pressure of NH_3 . Complete conversion of Ga to high-purity hexagonal GaN powder can be achieved within 6 h at 540 °C in 100 MPa NH_3 in the presence of 0.2 mol% NH_4I .

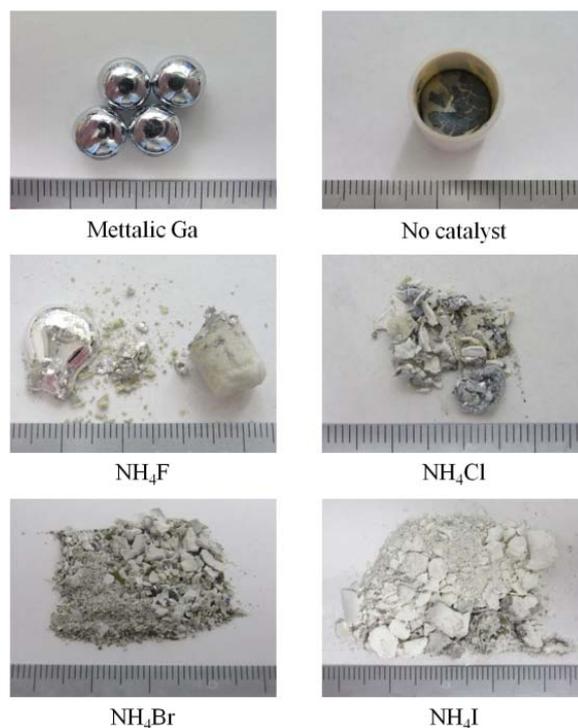


Figure 1. Photographs of Ga metal and as-synthesized GaN powders.

Impact of SiN_x passivation on the maximum current of AlGa_N/Ga_N HEMTs

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Dielectric films such as SiN_x, SiO₂, and Al₂O₃ are playing key roles in AlGa_N/Ga_N heterostructure field-effect transistors (HFETs) both as surface passivation and as gate insulating layers [1]. It has been observed by several groups, including ours, that thin SiN_x (<50 nm) deposition on AlGa_N/Ga_N HFETs by plasma enhanced chemical vapor deposition (PECVD) can significantly change the two-dimensional electron gas (2DEG) density (n_s). The origin of this change in n_s has not been carefully analyzed until now. The effect of SiN_x layer on AlGa_N/Ga_N HEMTs has been challenging since it is difficult to decouple the effects of the SiN_x induced-strain from changes in surface potential.

In this work, we have investigated the impact of SiN_x passivation in the transport properties of AlGa_N/Ga_N heterostructures grown on Si(111) substrates. In order to cancel the variation of the n_s introduced by the possible additional strain induced by the SiN_x layer, we have fabricated free-standing SiN_x/AlGa_N/Ga_N/SiN_x heterostructures (cf. Figure 2). These structures have allowed, for the first time, to study the intrinsic effect of passivation layer of SiN_x on the electrical characteristics of Ga_N-based devices. It was found that surface strain induced by the passivation layer is the main contributor to the change in the charge density in the Ga_N-based devices. A comparative study of tensile and compressive SiN_x dielectrics on AlGa_N/Ga_N grown on Silicon (111) shows an increase by ~13% and a decrease by ~40% in n_s, respectively (cf. Figure 1). These results pave the way to a new degree of freedom in the design of Ga_N electronic devices, local strain engineering.

This work has been partially funded by the MSD MARCO program and the ONR Young Investigator project, monitored by Dr. Paul Maki. Devices were fabricated at the MIT Microsystems Technology Laboratory and Raman characterizations were performed at the Harvard CNS facilities.

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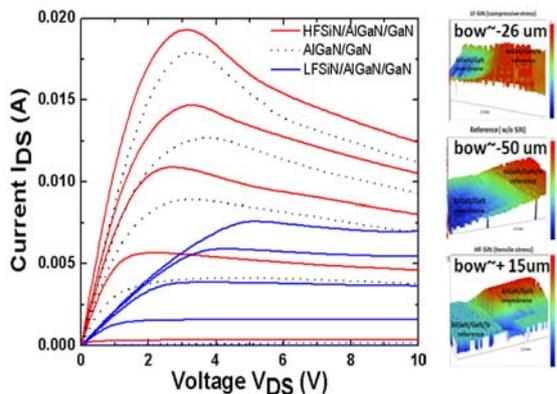


Fig.1. I(V) characteristics of AlGa_N/Ga_N, HFSiN/AlGa_N/Ga_N and LFSiN/AlGa_N/Ga_N.

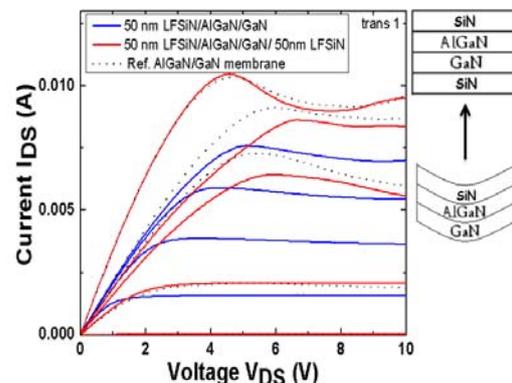


Fig.2. I(V) characteristics of AlGa_N/Ga_N, LFSiN/AlGa_N/Ga_N, and LFSiN/AlGa_N/Ga_N/LFSiN.

Growth temperature effect on two-dimensional electron gas density in AlGa_N/Ga_N heterostructure

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The two-dimensional electron gas (2DEG) density in the conventional AlGa_N/Ga_N heterostructure is mainly governed by the spontaneous and piezo-electrical polarization fields which can in turn be effectively controlled by the AlGa_N barrier thickness and composition. However, in our study, we have found that the growth temperature also has a significant influence on the 2DEG density.

A series of samples of 20 nm-thick Al_{0.25}Ga_{0.75}N/Ga_N heterostructures on 4H-SiC substrates with AlN nucleation layers were grown in a temperature range 1000 °C to 1060 °C by hot-wall MOCVD, and characterized by high resolution – X-ray diffraction (HR-XRD), photoluminescence (PL) measurements and mercury probe capacitance-voltage (C-V) measurements. All samples show very similar crystalline quality, surface morphology as well as AlGa_N composition and thickness. Nevertheless, C-V measurements reveal that the 2DEG density remarkably increases from 2.9E12 to 8.8E12 cm⁻² with increased growth temperature in the investigated range. Similarly, the sheet resistance of the samples reduces from 2200 to 378 ohm/sq, as-measured by contactless eddy-current sheet resistance.

Since all Ga_N buffer layers show extremely low capacitance at voltages beyond pinch-off voltage, one can expect that most of 2DEG is contributed from the AlGa_N layer or its surface. In order to gain further understanding of the growth-temperature-dependent 2DEG density phenomenon, we performed: secondary ion mass spectrometry (SIMS) on impurities in the AlGa_N layer, e.g. O, Si, C, H, as a function of growth temperature, deep level transient spectroscopy (DLTS) to study electrically active defects, and x-ray photoelectron spectroscopy (XPS) analysis of AlGa_N surface stoichiometry as well as Hall measurement and THz ellipsometry.

Our findings on the 2DEG density variation with growth parameter provides us with a new tool - in addition to scaling the barrier thickness, adjusting the Al content in AlGa_N layer or applying surface passivation - to understand and control the 2DEG in the AlGa_N/Ga_N heterostructure.

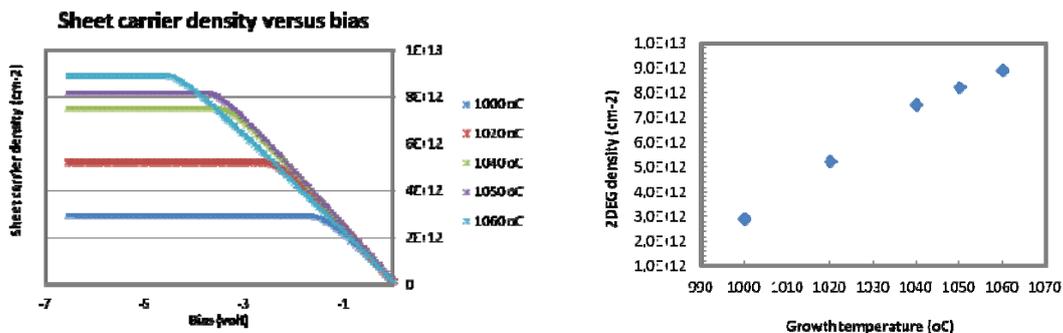


Figure 1. (a) C-V measured sheet carrier density versus bias for AlGa_N/Ga_N structure grown at different temperatures

(b) Growth temperature-dependent 2DEG distribution

Experimental and theoretical study of electrical, optical, and thermal processes in high-power InGaN/GaN LEDs

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Lateral current spreading is an important factor controlling performance of high-power III-nitride LEDs. While the current density distribution cannot be measured directly, it affects both the light emission and temperature non-uniformities in LED chips. In particular, the near-field emission intensity and temperature distributions are frequently believed to be rather similar to the vertical current density distribution in the active region, which is not generally the case. This study is aimed at detailed understanding of correlations between electrical, thermal, and optical processes in high-power LED chips by using a number of advanced experimental techniques and numerical simulations. Another goal of the study is to examine the current crowding effects on the overall chip performance, including the emission efficiency droop with current.

An InGaN/GaN heterostructure emitting light at 450 nm with short-period superlattice active region grown by MOVPE has been processed into 1300×1300 μm high-power flip-chip LEDs with highly reflective Ag-based *p*-electrodes [1]. Near-field emission intensity and temperature distributions in the active region were measured by mapping through optical and IR microscopes the LED chip images formed by its own electroluminescence and IR thermal radiation [2]. The spatial resolution of the measurements was ~3μm. In view of different emissivity of various chip units, the temperature was preliminarily calibrated by monitoring the IR emission at zero current in the temperature range of 20-100⁰C. The thermal resistance of the LED was estimated from the rate of forward voltage relaxation upon the stepwise current supply.

3D coupled simulation of the current spreading, heat transfer, and light extraction from the LED chip has been carried out with the SimuLED software [3]. The current density-bias characteristic of the LED heterostructure was approximated by the Shockley's model with the non-ideality factor of ~1.5-2.0, while the ABC-model with appropriate parameters accounted for the internal quantum efficiency dependence on the current density. Ray tracing determined the efficiency of light extraction from the LED with account of computed current density distribution in the active region.

We have found the current density to be distributed very non-uniformly inside the die even in the case of optimized flip-chip design: the maximum current density is typically ~2-3 times higher than its mean value. The near-field emission distribution qualitatively resembles that of the current density but with less steep variations, which is caused by the IQE droop at high current density. In contrast, the temperature is distributed more or less uniformly (the lateral temperature variation is ~25% of the overall overheating) due to intensive lateral heat transfer in the substrate and submount. For the chip design considered, the LED external quantum efficiency is found to be weakly sensitive to the current crowding. The simulation results agree well with the measurements.

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Peculiarities of III-Nitride heterostructure design and MBE growth conditions for two-dimensional electron gas and acoustic waves based devices

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A number of III-nitride devices have been already developed and are widely exploited, but high dislocation density in heterostructures, strong internal fields and related relaxation processes cause the great importance of stress control during epi-growth and planar processing. Here we report on ammonia MBE growth and heterostructure design solutions enabling lower dislocation density and good electron confinement in double-heterostructure field effect transistors (DHFET) or, alternatively, uniform in depth mechanical properties of AlN-based film bulk acoustic resonators (FBAR).

DHFETs oriented technology. Crystal quality of heteroepitaxial layers is improved with growth temperature rising being the necessary condition for lateral coalescence of the nuclei's grains [1]. Earlier we have shown [2] that growth temperature as high as 970°C and NH₃ flow of 400 sccm are beneficial for GaN NH₃-MBE. Similar tendency was observed in plasma-assisted MBE of III-nitrides, the technique known by its «classic» approach with moderate growth temperatures and V/III ratios close to unity [3]. At the same time, thermal stability of AlN is sufficiently higher which allows its growth at even higher temperature at the initial growth stage to improve coalescence of the nuclei's blocks. So, the main peculiarity of our approach for fabricating DHFET structures is using of rather thick AlN template grown at extreme for MBE substrate temperatures in the 1050-1200°C range. Being combined with proper AlGaN transition layers, such a design gives dislocation density well below than 10⁹ cm⁻² that is one of the best results reported for MBE-grown nitrides on mismatched substrates. 0.5-μm DHFET device processing is used to obtain power density of 4-5 W/mm in C-band. The *s*-parameters obtained from transistors with a 0.15 μm gate reveal $f_t \approx 60$ GHz and *MaxGain* of 10dB at 35 GHz, which paves a way towards millimeter-band applications.

FBARs oriented technologies. Since the frequency is inversely proportional to the film thickness, the resonant frequencies in acoustic wave resonators based on AlN from hundreds of MHz to tens of GHz can be realized [4]. AlN layers used so far in SAW and, until recently, BAW resonators intended for working bands less than 5 GHz could be deposited by various kinds of sputtering, which is quite simple, robust and relatively cheap in production. But to reach higher frequencies epitaxial growth methods providing more precise thickness and composition control were involved [5]. In contrast to growth process we used for DHFETs, we have developed alternative, stress cycling technique that allows growing AlN layers up to a few microns on Si substrate without any cracks, having a uniform thickness and XRD-confirmed crystal quality. A number of FBARs varied by AlN thickness and metal Bragg stacks design were successfully fabricated with resonant frequencies in the 1-8 GHz range, which correspond to theoretically predicted values.

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Manipulation of Carrier Distributions in III-Nitride Visible Light-Emitting Diodes by Hole Injection Layer and Electron Blocking Layer

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Effective carrier injection from p-type layer and transport through multiple-quantum-well (MQW) and following uniform distribution of electrons and holes are believed to be an effective way to minimize the efficiency droop. In the present study, we report on the behavior of electrons and holes transport and resulting distributions of the carriers in visible III-nitride (III-N)-based light-emitting diodes (LEDs) in relation to the effect of hole injection layers and electron blocking layers (EBLs). We introduced a triple-wavelength (TW)-MQW active region structure which has different indium (In) mole fraction in each $\text{In}_x\text{Ga}_{1-x}\text{N}$ QW in order to effectively trace the influence of the transport of carriers and resulting distributions for radiative recombination. In addition, Si doping was introduced in selected quantum-well barriers (QWBs) to intentionally control the carrier transport. LED epitaxial structures with various hole-injection layers and with and without EBLs were grown on c-plane (0001) sapphire substrates by metalorganic chemical vapor deposition in a Thomas Swan 6×2" reactor system. The LED structures consist of a 3- μm -thick Si-doped GaN layer with an electron concentration of $n \sim 5 \times 10^{18} \text{ cm}^{-3}$, a three-period $\text{In}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$ (2.5/12 nm, violet color for QW1, blue for QW2, cyan for QW3 in the sequence of the growth) MQW active region, a Mg-doped $\text{In}_{0.18}\text{Al}_{0.82}\text{N}$ EBL (or a Mg-doped $\text{Al}_{0.2}\text{Ga}_{0.8}\text{N}$ EBL or without an EBL), and Mg-doped p-GaN (or Mg-doped p- $\text{In}_x\text{Ga}_{1-x}\text{N}$) hole injection and contact layers. The electro-optical properties of TW-LED structures were characterized from $350 \times 350 \mu\text{m}^2$ fabricated LEDs. In the case of the LEDs with higher indium mole fraction in p- $\text{In}_x\text{Ga}_{1-x}\text{N}$, emission from QW1 becomes stronger. This gradually increased EL intensity of QW1 compared to QW2 and QW3 for the LEDs with increasing indium mole fraction in p- $\text{In}_x\text{Ga}_{1-x}\text{N}$ layer indicates that more holes can be transported to the lower QW by hole injection layers. The EL spectrum of the TW-LED without an EBL showed the highest emission peak at QW3, one closest to the p-type layer, and gradually decreased in QWs with increasing distance from the p-GaN layer for all injection current ranges. For the TW-LED with an InAlN EBL, however, QW2 has the highest emission peak intensity among the three QWs at low injection current and the emission from QW1 is also much higher than that of the TW-LED without an EBL. In addition, emission intensity of QW1 is comparable to that of QW3 at the injection current of 80 mA. To further investigate the carrier transport, we utilized Si doping in a selected QWB. The Si-doped QWB acts as a hole blocking layer so that the hole transport into lower QWs is hindered and correspondingly, most holes are confined at QW3. We will compare electroluminescence characteristics of TW-LEDs employing InAlN and AlGaN EBLs in comparison to one without EBLs and employing various p- $\text{In}_x\text{Ga}_{1-x}\text{N}$ layers in order to distinguish the carrier transport and distribution in the active region. This different carrier dynamics and related efficiency droop behavior will be further discussed.

Comparative study of $\text{In}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$ and $\text{In}_x\text{Ga}_{1-x}\text{N}/\text{Mg}_y\text{Zn}_{1-y}\text{O}$ High Efficiency Solar Cells

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$\text{In}_x\text{Ga}_{1-x}\text{N}$ materials are attracting great interest because of their potential in various applications, and mainly in optoelectronics, in blue-green light emitting diodes and lasers. $\text{In}_x\text{Ga}_{1-x}\text{N}$ materials revealed themselves also as good candidates in the field of high efficiency photovoltaic solar cells. This is related to the important properties which are shown by $\text{In}_x\text{Ga}_{1-x}\text{N}$. $\text{In}_x\text{Ga}_{1-x}\text{N}$ alloys exhibit direct energy gaps from Infra-red (0.65eV in InN) to Ultra-Violet (3.4eV in GaN), an energy interval which corresponds to the maximum solar energy spectrum. $\text{In}_x\text{Ga}_{1-x}\text{N}$ materials are also characterized by high optical absorption coefficients, high carrier mobilities and drift velocities, and excellent mechanical, thermal and radiation resistances.

$\text{In}_x\text{Ga}_{1-x}\text{N}$ solar cells were designed first as $\text{In}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$ p-n and p-i-n junctions. However, good quality devices were restricted to a narrow In-contents range $x < 10\%$. This is due to several restrictions inherent to $\text{In}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$ epilayer as a big lattice-mismatch between InN and GaN, piezo-electric polarization effects due to their non-centro-symmetric stable Würtzite crystal structure, as well as In-growth temperature restrictions and In-phase separation limit.

Substantial improvement of these structures could be achieved by using $\text{In}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$ multiple quantum wells (MQW) where In-phase separation could be overcome, and In-contents up to $x \sim 30\%$ could be introduced.

In this work, we perform a comparative computational study of $\text{In}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$ versus $\text{In}_x\text{Ga}_{1-x}\text{N}/\text{Mg}_y\text{Zn}_{1-y}\text{O}$ MQW solar cells. In both cases, we perform a detailed theoretical investigation as a function of x for $\text{In}_x\text{Ga}_{1-x}\text{N}$ and y for $\text{Mg}_y\text{Zn}_{1-y}\text{O}$ for various solar cells parameters as I-V characteristics, open circuit voltage, short circuit current, fill factor and external quantum efficiency.

We are interested more particularly in the effects of internal electric field due to spontaneous and piezoelectric polarizations which are found to limit the performance of the solar cells. In order to avoid these undesirable polarization effects, we investigate a nonpolar structure made of $\text{In}_x\text{Ga}_{1-x}\text{N}/\text{Mg}_y\text{Zn}_{1-y}\text{O}$ MQW grown on nonpolar ZnO substrate. Such a structure makes it possible to increase In-contents up to 35% in $\text{In}_x\text{Ga}_{1-x}\text{N}$ QW thanks to the fact that $\text{Mg}_y\text{Zn}_{1-y}\text{O}$ is lattice matched to $\text{In}_x\text{Ga}_{1-x}\text{N}$ at $x=17\%$ for $y=0$, and at $x=26\%$ for $y=20\%$.

The description of quantum states in nanostructures GaN / InGaN using the envelope functions. The role of boundary conditions

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The envelope functions method is widely applied for quantum modeling electron and hole states in heterostructures based on nitrides of In and Ga. Variants of the method differ from each other by a form and a contents of matching conditions of the envelope functions on heteroboundaries. It is necessary to emphasize that the continuity of envelope functions on heteroboundaries is postulated in most of works. The second group of conditions is determined by integrating the equations for the envelope functions in a small interval of coordinates, comprising the heterojunction. Let us name such matching conditions as the standard conditions. They can be justified when the contacting materials are chemically similar to each other and have similar crystal structure. However it is not always achieved even for related materials due, for example, to distinction of volumes of elementary cells in materials of heterostructures. Similar case is realized in heterostructures on the basis of nitrides of Al, Ga, In. It is of interest to find out a role of distinctions of Bloch functions in formation of matching conditions for envelope functions.

The present work devoted to model problem of analysis of quantum wells and superlattices based on GaN/In_xGa_{1-x}N(0001). The conditions of continuity of full wave functions and their derivatives are used; on this base the approached boundary conditions for envelopes are reduced. For this purpose a matching matrix for the wave functions on heteroboundary GaN and the solid solution In_xGa_{1-x}N are calculated using the method of scattering matrix by making use of model pseudo-potentials. Using the technique developed by us earlier, from elements of a matching matrix for the wave functions we have found the matching conditions on heteroboundary for actual envelope functions. In a similar approach the matching conditions of envelope functions have discontinuity on heteroboundary. Moreover envelope functions and their derivatives are mixed at the heterojunction thus that these can not be separated.

The numerical values of the parameters of mixing functions and their derivatives depend on a unit of measurements of a length. We have considered hole waves which move normally to heteroboundary by making use both the standard matching conditions and our conditions formulated above. We calculated the energy levels and the envelope functions for holes in quantum wells and superlattices. For illustration we present results obtained for the case of $x = 0.2$. The widths of the barriers and wells equal to 2 nm. We restrict our discussion to case of the hole waves propagating along the [0001]. It turned out that discontinuities of envelope functions at heterointerfaces are of small magnitude. It was found that for two different matching conditions the calculated energy levels differ by a small value of 1-4 meV. The corresponding envelope functions are similar on the whole. But in the case of the standard version these have symmetry about the center of the quantum well, whereas the symmetry is absent in the case our matching conditions. This qualitative result is consistent with the symmetry of the structure and emphasizes the validity of our suggested matching conditions. The analysis of the electron and hole states in superlattices and quantum wells based on heterostructures with a different choice of a solid solution of InGaN was carried also.

Selectively-sensitive MSM-photodetectors based on AlGaN/AlN and ZnCdS/GaP heterostructures

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Sunlight is strongly absorbed in the atmosphere at the wavelengths < 300 nm while almost freely reaching the Earth's surface at 300-450 nm and is of growing attention for both health and military applications. Sensitive detectors for these wavelengths are of special interest. The metal-semiconductor-metal (MSM) photodetector has become an attractive device in modern optoelectronics. We report on the growth, fabrication and characterization of high-speed MSM-detectors based on AlGaN/AlN and ZnCdS/GaP heterostructures. The heterostructures were grown by MOVPE, the optimum growth temperature was found to be 1050^oC for AlGaN/AlN samples on sapphire substrates and 415^oC for ZnCdS samples on s.i.GaP. The surface roughness has been monitored by atomic force microscopy. Optical properties and bandgap energies of epilayers were characterized by PL and photoreflectance measurements. After the film growth Schottky barrier interdigital contacts were evaporated to form MSM-diode structures. The finger width and gap were 2.8 μm and active diode area 100x100 μm^2 thus suggesting short transit times of the carriers, low capacitance of the diode and high-speed operation of the detectors.

The AlGaN/AlN MSM-diode consists of 110 nm AlN filter layer on sapphire substrate, thin Al_{0.8}Ga_{0.2}N isolating layer and 300 nm Al_{0.6}Ga_{0.4}N active layer with the interdigitated Au/Ni contacts on top of it. The spectral responsivity of AlGaN/AlN MSM-structures demonstrates the ability of these detectors for true solar-blind response with maximum current responsivity ~ 0.1 A/W at 240 nm under illumination from substrate. The I-V measurements showed low dark currents of ~ 50 pA at 40 V bias. Junction ideality coefficient in the range of 1.1-1.20 indicates a good quality of Schottky barriers in the AlGaN MSM-interdigitated contact system. Detector demonstrates sharp spectral selectivity, at 240 nm the FWHM of detector response is equal to 46 nm.

The significant advantage of ZnCdS compound is that it can be isoperiodically grown on the GaP substrates with low level of dislocations thus reducing dark current of the diode. The I-V measurements in this system showed very low dark current of 2 pA at 40V. Spectral response of the ZnCdS/GaP MSM-diodes was characterized under various bias conditions. Detectors demonstrate strong dependence of spectral response on bias voltage. For the bias up to 60 V electric field concentrates mainly in the upper ZnCdS thus implying maximum response at 360 nm whereas for 80-100V penetrates deeper into GaP substrate thereby broadening detector spectral response and shifting diode maximum sensitivity at 440 nm. For low bias the 360 nm to 440 nm rejection ratio is equal to 190 while for 100 V bias this ratio is nearly 1. Thus the cutoff wavelength of the ZnCdS/GaP MSM-detector shifts from 360 to 440 nm and detector is selectively sensitive with changing bias. One of the practical applications of these ZnCdS/GaP detectors is a control of near UV-radiation dangerous for human being.

In conclusion, while ZnCdS/GaP detectors result in lower dark currents, MSM-diodes on AlGaN/AlN allow realizing more short wave UV selective response of the detector.

C-doped asymmetric cubic AlN/GaN multi quantum well structures for high electrical isolation to 3C-SiC substrates

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AlGa_N/Ga_N hetero-junction field-effect transistors (HFETs) are presently of major interest for use in electronic devices, in particular for high-power and high-frequency amplifiers. This is motivated by their potential in commercial and military applications, e. g. in communication systems, radar, wireless stations, high-temperature electronics and high-power solid-state switching. Currently, state of the art HFETs are fabricated of the *c*-plane surface of wurzite (hexagonal) AlGa_N/Ga_N hetero structures. Their inherent spontaneous and piezoelectric polarization fields produce extraordinary large sheet carrier concentrations at the AlGa_N/Ga_N hetero-interface. Therefore, all these devices are of the normally-on type. However, for switching devices and digital electronics field-effect transistors with normally-off characteristics are desirable.

A direct way to eliminate this polarization fields is the growth of non-polar cubic AlGa_N/Ga_N. The most suitable substrate for the growth of cubic AlGa_N/Ga_N hetero-structures is free standing 3C-SiC (001) with an effective lattice mismatch of 3.5%. However, the critical issue in the HFET operation on this substrate is its high conductivity, which increases the parallel conductance of the device resulting in a buffer leakage current. This is undesirable for high power and low noise applications and severely reduces the device performance. One way of the electrical separation of the active device from the substrate is an incorporation of *c*-AlN barrier layers, due to a large conduction band discontinuity between *c*-Ga_N and *c*-AlN of 1.4 eV [1]. Another possibility is the carbon doping of cubic Ga_N, at a certain carbon flux the conductance reduces by more than two orders of magnitude [2].

In this work we present a novel method of the electrical separation of the substrate and the HFET device. For the epitaxial growth of our structures free standing 3C-SiC (001) substrates with a free carrier concentration of $n=2 \times 10^{18} \text{ cm}^{-3}$ and a resistivity of $\rho=6.6 \text{ m}\Omega\text{cm}$ were used. On a 50 nm *c*-Ga_N buffer layer an asymmetric quantum well (QW) structure consisting of 10 cubic Ga_N:C asymmetric (1 nm/2 nm) QWs with 3 nm *c*-AlN:C barriers is grown. A 50 nm *c*-Ga_N cap layer completes the sample structure. The structural properties were analyzed by atomic force microscopy and high resolution x-ray diffraction. The samples were optically characterized by photo- and cathodoluminescence spectroscopy. Current-voltage measurements of these structures showed an increase of the serial resistivity by six orders of magnitude compared to an unstructured *c*-Ga_N reference sample.

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Nondestructive method for evaluation of electrical parameters of AlGaIn/GaN HEMT heterostructures

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The AlGaIn/GaN HEMT heterostructures are attractive for numerous device applications. Nitrides heterostructures are one of the leading candidates for high frequency applications from GHz up to THz range. Because of high cost of the technology, it is essential to use nondestructive characterization techniques for quick verification of the quality of fabricated AlGaIn/GaN heterostructures and prediction of the performance of microwave HEMT transistor based on measured characteristics of the heterostructures.

The frequency dependent capacitance and conductance measurements of Schottky contact characteristics supplemented with appropriate modeling i.e. impedance spectroscopy methods offers such possibility. It allowed direct evaluation of electrical parameters of the AlGaIn/GaN heterostructures by analyzing their experimental data using a proper equivalent circuit model.

The article presents the results of application of nondestructive impedance spectroscopy methods for the evaluation of electrical parameters of MOVPE AlGaIn/GaN heterostructures. The research carried out at WEMiF WrUT was devoted to optimization of the performance of microwave HEMT transistors. The obtained results will be presented and discussed. Two-column mercury probe with co-axial circle shaped contacts was used for the measurement. The axial symmetry of the contacts allowed us to apply the equivalent circuit model with distributed elements that includes an internal measuring contact with the diameter of 1 mm and an external contact of much larger area. The equivalent circuit includes the sheet resistance of 2DEG channel under the mercury contacts and between two contacts, as well as the junction capacitance. The fitting of the equivalent circuit elements to the measured impedance spectra of the mercury probe - AlGaIn/GaN heterostructure system, performed in a wide range of frequencies (20Hz - 13MHz) at different values of reverse bias, allowed us to determine the channel sheet resistance between the contacts and the channel resistance changes resulting from the change in 2DEG concentration, caused by bias change of the measuring contact. As a result, the 2DEG concentrations and differential drift mobility of the tested AlGaIn/GaN heterostructures were obtained without the need of test structures fabrication.

Additionally, the performed measurement enabled the estimation of the resistivity of the buffer and nucleation layers and the study of relaxation phenomena caused by deep levels and surface states. The obtained results were correlated with the growth parameters of the MOVPE process used for the fabrication of the AlGaIn/GaN heterostructures. They, together with the Hall measurement results, can be applied for prediction of HEMT transistors parameters.

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Determination of recombination lifetimes in InGaN/GaN based LEDs by differential lifetime analysis

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All indium gallium nitride/gallium nitride (InGaN/GaN) quantum well (QW) based light emitting diodes (LEDs) exhibit an efficiency droop, *i.e.*, reduction of efficiency at high operating current density. Although III-N materials have been extensively studied, there is no widely accepted single explanation for the droop. [1] Several reasons for this behavior have been proposed, such as density-activated defect recombination (DADR) [2], enhanced Auger recombination [3], electron leakage [4], and a unified model combining different mechanisms [1]. A common feature in all the proposed models is the reduction of radiative recombination efficiency at high current density.

Differential carrier lifetime analysis can be used to measure the recombination parameters of charge carriers in InGaN/GaN LED structures. [5] During the measurement the LED is injected with a direct current (DC) modulated by a small sinusoidal alternating current (AC) component. The phase difference between the light output and the input current can be analyzed by using the small signal rate equation model to obtain the recombination lifetimes of the carriers. As the carrier density is mainly governed by the DC component, the method enables measuring the recombination lifetimes as a function of carrier density by changing the DC component.

In this work the differential carrier lifetime analysis method is evaluated for determination of the carrier lifetimes of blue emitting InGaN/GaN QW LEDs. The modulation frequencies used in the measurements ranged from 10 kHz to 1 MHz. The electroluminescence was measured with a high-bandwidth silicon detector. The recombination lifetime was found to decrease from 450 ns to 220 ns when the current density was increased from 0.07 A/cm² to 5 A/cm². The modulation amplitude in the light output was found to noticeably decrease when the modulation period was comparable to the recombination lifetime.

Our results are in reasonable agreement with carrier lifetimes obtained from c-plane single InGaN QWs with time resolved photoluminescence (TRPL) [6]. In contrast to TRPL the differential lifetime analysis method allows a straightforward way to determine the carrier lifetimes as a function of excitation. The effect of current density on the radiative and non-radiative recombination lifetimes in an InGaN/GaN LED is evaluated on the basis of the obtained recombination lifetimes, efficiency measurements and simulations.

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High signal-to-noise ratio visible-blind photodetector by using slightly Mg-doping InGaN

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Visible-blind photodetectors working in the wavelength lower than 390 nm have big potential in the biomedicine, environment monitoring, photolithography, flame detection, etc. III-Nitride semiconductor InGaN is considered to be one of the best candidates for visible-blind photodetectors working in this region due to its high theoretical responsivity, high breakdown voltage, sharp cutoff wavelength, and expected low intrinsic noise. However, InGaN-based devices suffer from a high leakage current due to the high-density dislocations and high *n*-type background carrier concentration, which will result in very high noise during detection.

In this presentation, we will report a high signal-to-noise (SNR) ratio ($\sim 10^5$ @ 2V) visible-blind photodetector by using a slightly Mg-doping InGaN film. The crystalline quality of the InGaN film can be maintained by slightly Mg doping, while the strong *n*-type background can be compensated, resulting in a great decrease in the leakage current by nearly seven orders of magnitude at the bias voltage of 2 V.

Bis(methylcyclopentadienyl)magnesium was used as the Mg source during the deposition of InGaN, and the flow rate was kept at 0.2 $\mu\text{mol}/\text{min}$. The InGaN film was confirmed to be *n*-type by using hot probe station measurement. X-ray diffraction rocking curve revealed a full-width at half maximum of around 390 arcsec for (0002)-plane, which showed a high crystalline quality. Interdigital metal-semiconductor-metal photodetectors were fabricated with Ni/Au as the Schottky contacts.

The device showed an ultra-low leakage current of 9.9×10^{-13} A at 2 V, which is nearly seven orders of magnitude lower than the device fabricated on InGaN film without doping (8.9×10^{-7} A), as shown in Fig. 1. The dramatic reduction in the dark current brings forward a significant improvement of the SNR ratio (10^5 at 2 V) when the photodetector was illuminated by 378 nm (ultraviolet (UV)) light. It should be noted that the photodetector fabricated on slightly Mg-doping InGaN also showed a fast response speed and a high discrimination ratio. We observed no photoconductivity effect when mechanically measured by the current meter through shutting on/off the UV light. The discrimination ratio between 378 nm and 630 nm is nearly five orders of magnitude, which is greatly improved compared with device fabricated on InGaN (10^2) (Fig. 2). Such a high value guarantees the application of the present devices as the visible-blind photodetectors. The excellent UV/visible discrimination ratio and high SNR ratio are considered to be benefited from the marked decrease in the dark current by using a slightly Mg-doping InGaN film.

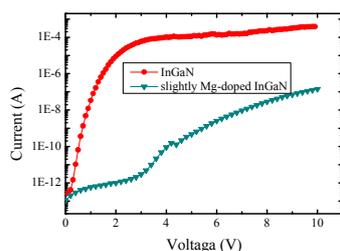


Fig. 1

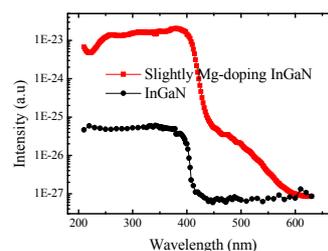


Fig. 2

Extended defects system as a main source of non-radiative recombination in InGaN/GaN LEDs

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Non-radiative recombination in blue InGaN/GaN power Light Emitting Diodes (LEDs) with multiple quantum wells has been a main point for a number of studies for more than ten years. However, it has not been clarified until now, whether non-radiative losses in these structures are caused by *point defects* or by *extended defects system (EDS)* piercing all layers of LEDs. The aim of this paper is to clarify the relative contribution of extended and point defects in non-radiative recombination processes in InGaN/GaN LEDs.

Comprehensive investigations of the processes were performed using *low frequency noise measurements* in the frequency range from 1 Hz to 10 kHz and current density diapason 10^{-4} - 50 A/cm², *current-voltage dependencies* in the range from 10^{-13} to 1 A at temperatures 300 – 400 K, and *external quantum efficiency dependencies* on current density j . Set of InGaN/GaN LEDs with different active region design and different maximum external quantum efficiency over the range 25 % to 45 %.

The properties of EDS had been studied earlier. EDS includes threading dislocations, their accumulations and numerous dilatation and dislocation boundaries. These peculiarities depend strongly on grown conditions (grow modes 2 or 3, buffer layers, etc.) and determine numerous forms of nanostructural arrangement with different degree of order and leakage current values at $U < 2$ V [1].

The dependencies of spectral noise density S_I and S_V on j in LEDs with different leakage current values showed a correlation between the noise level and the degree of order. The S_I and S_V dependencies demonstrate more complicated behavior in comparison with analogous dependencies in traditional A³B⁵ LEDs. The shape of observed S_V dependences shows non uniform distribution of injection current and intensification of this phenomenon with the leakage current values augmentation. $S_I(j)$ dependences reflect changes in the defect system properties under injection current and the behavior of this system differs noticeably from one of point defects. The significant increase in S_I values as well as $S_I \sim j^3$ was observed at $j > 10$ A/cm². These peculiarities, observed EQE(j), $S_V(j)$ dependences conflict with point defects approach. The local development of nonradiative process took place in EDS. Thus EDS is the main source of non-radiative recombination in blue power InGaN/GaN LEDs.

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Mechanisms behind efficiency droop and degradation in InGaN/GaN LEDs

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The solid state lighting programs based on blue power InGaN/GaN LEDs develop in many countries. However, there are two fundamental problems on the way to reasonable cost of Lumen per Watt: droop of the external efficiency occurring at the injection currents as low as 10 A/cm² and broad distribution of the lifetimes of LEDs with close parameters. Though origin of these troubles has been discussed for more than a decade, final agreement is still not achieved. Generally accepted approach to consider these problems is based on the conventional Shockley-Read-Hall (SRH) defect recombination scheme neglects complicated defect structure of InGaN/GaN LEDs. Some authors [1] drew attention to major to nonradiative process out of scope SRH model. Different mechanisms taking into account system of the extended defects (EDS) are desirable and discussed in this paper.

In order to investigate nature of the mentioned phenomena of efficiency droop and ambiguous degradation we have carried out complex study including electrical and optical measurements at different stages of degradation of a series of InGaN/GaN LEDs with different maximum efficiency in the interval from 25% to 45%. Besides usual measurements of the external quantum efficiency and I-V characteristics, we have studied low frequency noise power spectral density S_I and S_V as a function of current at the frequency range from 1 Hz to 10 kHz and injection current densities j from 10⁻⁴ to 50 A/cm².

The properties of EDS piercing all the LED layers and forming high conductivity paths as well as the correlation ones with leakage current at $U < 2$ V and the values of S_I had been earlier clarified. It has been found out that $S_I(j)$ and $S_V(j)$, dependences for InGaN/GaN LEDs are more complicated than for traditional A³B⁵ materials, and significant increase of the values of S_I as well as appearance of the regions where $S_I \sim j^3$ at $j > 10$ A/cm² related to local change of EDS properties under injection current together with EQE droop, were observed. The shape of observed $S_V(j)$ dependencies confirms non-uniform distribution of injection current and intensification of this phenomenon with the leakage current increase. These results shed light on the EDS contribution in efficiency droop. Appearance of $S_I \sim j^4$ at $j > 10$ A/cm² at different stages of LED aging was observed as well as the same dependencies at $j \leq 10^{-2}$ A/cm² for LEDs under the change EQE for more than twice. Moreover, both non-uniform distribution of the injection current and leakage current values at $U < 2$ V of LEDs increase during the process of aging. These peculiarities indicate presence of the very strong local overheating along EDS increasing with injection. The conductive paths related to EDS evoke crowding effect like the one related to poor geometry of the contacts. Apart from this, it is made clear that fast development of degradation process is related to redistribution of In at local fluctuations in the InGaN alloy compositions under injection. Thus both fundamental problems hampering current progress of the solid state lighting have common nature related to EDS, playing a key role in these nanostructured materials.

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Capacitance-Voltage and Admittance Investigations of InGaN/GaN MQW LEDs: Frequency Dependence

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Last years InGaN/GaN heterostructures are widely used for light emitting applications. In spite of a considerable amount of effectively working devices on GaN-based structures, some problems concerning material quality and possibilities of its improvement still remain. In particular, the small enough diffusion length observed in these materials implies that the concentration of recombination centers should be rather high (about real doping concentration). Nevertheless, such centers were not directly observed and their origin still unclear. Dislocation density registered by electron-beam-induced-current technique and concentration of deep traps observed by deep level transient spectroscopy [see, e.g., 1] are, as a rule, two or three times lower than it is needed to explain such recombination.

For an investigation of essential changes in concentration of electrically active defects, the capacitance-voltage (C-V) profiling and other impedance techniques (e.g., admittance spectroscopy) are informative and convenient. However, in spite of wide usage of these techniques for characterization of InGaN/GaN structures [e.g., 2,3], an interpretation of the measurement results is often misapprehended due to the complexity of the structures. For example, in [4] it was shown that the commonly used conditions for C-V profiling of InGaN/GaN multiple-quantum-well (MQW) structures correspond to an intermediate case between low- and high-frequency capacitance approximations that could prevent the correct extraction of the depth value from the C-V data. It was demonstrated also that the profile of an apparent carrier distribution obtained from C-V measurements “shifts” from the p-n junction edge with temperature and frequency, that is obviously impossible and needs adequate explanation [4].

In the presented work, the InGaN/GaN MQW LED structures with five QWs grown by MOCVD technique on sapphire substrate are experimentally investigated in wide temperature and frequency ranges. From the study of C-V curves and impedance-frequency dependences, it is shown the existence of, at least, two essential steps in the imaginary part of the impedance over the investigated temperature-frequency range. Possible origin for these features and the contributions of such elements of the considered system like deep traps, carriers in the QWs, etc. are discussed. Using computer simulation, an analysis of possible influence of these contributions on the results of C-V and admittance measurements is carried out.

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Ab initio modeling of AlN/GaN multi quantum wells

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A new method of determination of the physical properties of GaN/AlN multi-quantum well system used direct modeling of single well-barrier segment by density functional theory (DFT) quantum mechanical calculation with periodic boundary conditions for electric potential [1]. Using this formulation it was proved the existence of not only the polarization induced charge but also polarization induced dipole layer at GaN/AlN interface. Multi-quantum well systems were modeled using DFT calculations. From these results, the electric field, polarization charges and polarization dipoles within the well-barrier structure, were obtained. The dependence of the physical properties of the wells on their thickness and also on the thickness of the barriers was elucidated. It was shown that these fields depend critically on the well-barrier thickness ratio. The field gives rise to emergence of Quantum Confined Stark Effect (QCSE), changing the energies of quantum states of the electrons and holes in the wells and also separating the wavefunctions of both quasiparticles in space. The overlap of these functions is calculated, showing considerable separation in space which significantly reduced the oscillator strength of the optical transitions, and consequently, the optical efficiency of nitride based light emitting diodes (LED) and laser diodes (LD).

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p-type AlInN for Lattice-Matched Nitride Devices

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The AlInN alloy can be grown perfectly lattice-matched to GaN for an In composition of around 18% at which it has a larger bandgap energy and a high refractive index contrast with respect to GaN [1]. Lattice-matched AlInN/GaN structures have already shown a variety of applications, such as near-infrared intersubband transition devices [2], high-reflectivity distributed Bragg reflectors [3], and high optical confinement LDs [4]. In addition, AlInN offers a wide tuning capability of its bandgap energy from 6.0 eV (AlN) to 0.7 eV (InN). Therefore, AlInN-based lattice-matched structures should allow us to use the full potential of III-nitride semiconductors by increasing the design flexibility for high-performance LEDs and LDs from ultraviolet to infrared.

Intentional p- and n-type doping of AlInN is crucial for the AlInN-based devices. However, undoped AlInN has shown n-type conduction with high residual donor concentrations of $10^{18} - 10^{19} \text{ cm}^{-3}$, which is a significant obstacle to p-type doping. Although Mg-doped AlInN has been employed as a cladding layer or an electron blocking layer in LEDs [5, 6], p-type conduction of AlInN has not been reported. In this work, we demonstrate p-type AlInN by Mg doping and study its doping mechanism.

A series of Mg-doped AlInN layers with different Mg concentrations [Mg] was grown on sapphire (0001) substrate by MOVPE. The sample (LED structure) consisted of Mg-doped GaN, 100-nm-thick Mg-doped AlInN, InGaN/GaN MQWs, and Si-doped GaN. To reduce the residual donor concentration for p-type doping, AlInN was grown under In-rich condition to benefit from the In surfactant effect. After growth, samples were annealed in N₂ to activate the Mg acceptors. Coherent and single-phase growth of Mg-doped AlInN was confirmed by x-ray diffraction measurement.

p-type conduction of Mg-doped AlInN was confirmed by capacitance-voltage (C-V) measurements. The depth profile of the net acceptor concentration ($N_A - N_D$) was uniform in the Mg-doped AlInN layer. As the [Mg] increased to $2 \times 10^{19} \text{ cm}^{-3}$, the $N_A - N_D$ increased to $5.3 \times 10^{18} \text{ cm}^{-3}$, confirming that Mg acts as an acceptor in AlInN. On the other hand, the $N_A - N_D$ is still lower than [Mg], indicating that most of Mg acceptors are compensated. At high [Mg] $> 2 \times 10^{19} \text{ cm}^{-3}$, the $N_A - N_D$ decreased with the [Mg], implying that additional compensating defects are generated. For Mg-doped AlInN, one of the compensating defects is related to surface pits, called V-defects, because higher $N_A - N_D$ was obtained for samples with lower pit densities and the pit density increased with the [Mg]. At the facets of the pits, oxygen, which compensates the Mg acceptor, is known to be preferentially incorporated.

Finally, to demonstrate the feasibility of p-type AlInN in devices, characterization of LEDs was carried out. LEDs showed strong electroluminescence (EL) from the MQWs, attesting the hole injection from the p-type AlInN layer to the MQWs. Thus, the p-type doping of AlInN will open a new possibility for lattice-matched nitride devices.

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Optical and electrical characteristics of AlGaN-based UV LEDs

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The main drawback of AlGaN-based UV LEDs is their low external quantum efficiency (EQE). Therefore, it is important to determine key mechanisms of efficiency droop. For this purpose a comprehensive study of the optical and electrical characteristics of the UV LEDs manufactured from the epitaxial structures grown by Nitride Crystals was performed.

The UV LED epitaxial AlGaN/GaN double-heterostructures are entirely grown by chloride-hydride vapor-phase epitaxy (CHVPE). Each heterostructure consists of an undoped grading AlGaN layers (buffer layers), n-AlGaN hole-blocking layer, n-AlGaN light-emitting layer, p-AlGaN electron-blocking layer and p-GaN top layer. Diced individual LEDs have the dimensions of 310 μm \times 310 μm .

The measurements of spectral characteristics were carried out using a stand that was developed on quick scanning HR4000 spectrometer manufactured by Ocean Optics and comprising an integrating sphere. The stand allows us to study the behavior of radiation characteristics in a wide range of direct currents (10^{-8} – 0.2 A), both in constant-current and pulsed-current mode operation, to determine the temperature of the LED active region as well as the current-voltage (I-V) characteristics of devices.

The peak wavelength of the samples was in the range of 360-365 nm with a FWHM of about 10 nm at operating current (20 mA). It was determined that with a current increase the spectral maximum shifted towards longer wavelengths and a considerable spectral broadening occurred. The power-current (P-I) characteristics significantly deviated from the linear dependence at the current of more than 30 mA and at currents of 80-100 mA the power started to decrease. This indicates a strong decrease in the EQE at 30 mA current and higher.

For a more detailed study we performed the investigation of temperature characteristics of UV LEDs. It was found that the temperature of the active region increased to 150° C and higher at 100 mA current. Investigation of performance parameters revealed a significant decline of intensity at the current of more than 100 mA. We suppose that in the investigated samples the influence of self-heating effects on the intensity decline and EQE droop played a decisive role. However, Auger recombination may also contribute to the droop.

The degradation testing of the samples shows the lifetime exceeding several thousand hours at operating current.

Advantage of tapered and graded AlGa_N electron blocking layer in InGa_N-based blue laser diodes

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We report on the performance enhancement of InGa_N-based blue laser diodes (LDs) with tapered and graded AlGa_N electron blocking layer (EBL). The influence of such EBLs on hole injection and electron overflow effects is studied theoretically. Schrödinger-Poisson self-consistent method together with transfer matrix method was applied to calculate carrier distribution and transport properties in conventional EBL, tapered EBL and graded EBL. The influence of such EBLs on two dimensional waveguide modes is also considered.

The energy band profile for conventional structure exhibits an evident suppression of conduction band edge at the interface between the last quantum barrier (QB) and EBL. It was found that the polarization mismatch would introduce a fixed charge at this interface and create a parasitic electron inversion layer [1]. However, the strain-induced polarization charge would be spatially distributed due to the graded Al composition for both tapered structure and graded structure. More importantly, p-type doping of the original QB will help to lower the electron quasi-Fermi level with respect to conduction band edge and the parasitic electron inversion layer disappears.

Then transfer matrix method was applied to study the reflecting probability for electrons and transmitting probability for holes. The results indicate that the new structures favor the tunneling of low energy holes (below 500 meV) from the p-side to the active region. Meanwhile, more uniform carrier distribution and better balance between electrons and holes are obtained by proper modification of band diagrams.

In order to further investigate the influence of the above EBL structures on electron overflow effect in LDs, we performed advanced device simulation with LASTIP [2]. Suppression of electron overflow shows that the tapered and graded EBL are also efficient electron blockers. Threshold current density decreases from 2.0 kA/cm² for conventional structure to 1.6 kA/cm² and 1.4 kA/cm² for LDs with tapered and graded EBL respectively. The reduction of threshold current is resulted from the more uniform local gain profile, which eliminates the parasitic absorption in the quantum well near the n-side. Light output intensity at 100 mA shows a 15% and 22% increase for LDs with tapered and graded EBL respectively. It is noteworthy that new EBLs also increase the optical confinement factor slightly (about 3%). The performance of LDs can be further enhanced for high output power and low forward voltage by optimized EBL.

In summary, the tapered and graded EBL in InGa_N-based blue LDs would improve hole transport while not deteriorating the effect of electron confinement. As a result, Reduction of threshold current density and increase of light output power were also obtained. The above EBLs also offer a degree of freedom in refractive index design.

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Band modification in InGaN/GaN multiple quantum wells sandwiched by symmetrical thin low indium-InGaN layers

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High efficiency InGaN/GaN quantum wells sandwiched by symmetrical thin low indium InGaN (LI-InGaN) layer are grown by metal-organic vapor phase epitaxy. The LEDs with such MQW structure were investigated by high resolution X-ray diffraction (HR-XRD), high resolution transmission electron microscopy (HRTEM) and electroluminescence (EL). The light output power of such LEDs increases 45% compared to that from reference LEDs without the thin layer. The blueshift in the emission wavelength decreases. Moreover, current density at which peak efficiency increases and the efficiency droop improved by 106%. HRTEM images of the QWs sandwiched with LI-InGaN shows higher quality and less strain compared to the reference samples. After the simulation by the APSYS package of Crosslight software, the band structure and the carriers distribution in MQWs were obtained. The better performance of LEDs incorporating the insert layers is attributed to suppressed polarization from piezoelectric fields, high crystal quality and flatter band structure.

Fabrication and characterization of 80 μ m-thick free-standing homoepitaxial GaN-based blue LED

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A simple method of fabricating high power 80 μ m-thick free-standing homoepitaxial GaN-based blue LED was proposed and realized by metal organic chemical vapor deposition (MOCVD), hydride vapor-phase epitaxy (HVPE) and laser lift-off (LLO) technologies. Its advantage was demonstrated from chip fabrication process and chip structure. It was investigated by high resolution X-ray diffraction (XRD), high resolution transmission electron microscopy (HRTEM) photoluminescence, current-voltage and light output-current measurements. The width of (0002) reflection in XRD rocking curve was 173 arcsec for the free-standing LED, which was less than 258 arcsec for the conventional one. The HRTEM images show that the multiple quantum wells (MQWs) in 80 μ m-thick free-standing LED have a higher crystal quality. The light output was one time higher for the free-standing LED at 350mA and the forward bias was lower than that for conventional one. This high performance of 80 μ m-thick free-standing LED depends on the high crystal quality and strain relaxation in quantum wells. However, although the intensity of MQWs emission in PL spectra was doubled, both the wavelength and the width of the emission for the free-standing LED were increased. It could be attributed to the strain relaxed 80 μ m thick GaN, which would easily introduce indium composition and lead to InGaN phase separation.

Growth kinetics of III-nitrides by MOVPE and MBE

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GaN based optoelectronic devices such as light emitting diodes and laser diodes (LD) are widespread and commonly produced by metal organic vapor phase epitaxy (MOVPE). However, to grow structures with InN containing alloys, like InGaN or AlInN, the growth temperature needs to be reduced to decrease indium desorption and allow indium incorporation. This is especially important for achieving green emission in LDs, where an indium content above 25% is required [1]. With these conditions, the temperature approaches the usual temperature of GaN grown by ammonia molecular beam epitaxy (NH₃-MBE) where kinetics start to dominate the growth processes and need to be taken into account.

Here, we investigate the kinetic limited growth of GaN with both MOVPE and NH₃-MBE, and show that characteristic surface morphologies can be obtained by both techniques. Note that similar growth temperatures (close to MOVPE InGaN quantum well (QW)/barrier conditions) are used for both techniques. To limit the impact of dislocations on the surface morphology, free standing GaN substrates have been utilized. By thermal annealing standard MOVPE grown GaN layer morphology can be recovered, showing that those features are due to kinetic limitations.

Starting from a MOVPE GaN buffer layer and modifying the growth conditions, we demonstrate self assembled periodic structures of GaN on GaN. Those periodic patterns can be transferred to InGaN QWs deposited on top, leading to lateral variations in the QW which have been observed by cathodoluminescence [2] or micro-photoluminescence [3] by several groups. So far these observations have been tentatively attributed to originate from the InGaN [2]. We show how the morphological features can be controlled or suppressed and discuss the underlying physics. These features are likely due to an asymmetry originating from a Schwoebel barrier [4]. This effect is present in the III-nitrides, but only plays a role for low growth temperatures (e.g. at MOVPE QW/barrier or at NH₃-MBE GaN conditions) and is suppressed in plasma-assisted MBE by the use of a metal surface bilayer [5]. Finally, the implications on the growth of low temperature layer and for QW property optimization will be discussed.

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Gas phase mass transport and surface diffusion in extreme lateral overgrowth for cantilever sensor applications

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Nitride cantilevers can operate either as a piezo-resistor with a single layer of nitride or as high electron mobility transistors (HEMT). To date, these cantilevers have been fabricated from nitride-on-silicon materials by removal of the silicon substrate. However, the residual strain and high-density of dislocations may degrade their performance and make the calibration inconsistent. Thus, epitaxially lateral over-grown (ELOG) GaN devices are advantageous, as they have a low dislocation density and are basically strain-free. One technical challenge is that the ELOG wings on each side of the beam should be at least seven microns in width for a robust cantilever since reliable contacts must be fabricated and there is a need to remove the non-overgrown GaN by standard photolithography to fully release a cantilever. As a result there we need significant surface and gas phase diffusion [Mitchell et al, J. Cryst. Growth **222** (2001) 144) to enhance the lateral overgrowth rate.

ELOG bars of $>25\mu\text{m}$ in width have been obtained on GaN/sapphire templates with SiN_x masks (opening width~ 10microns). It should be noted there is significantly reduced parasitic growth within $150\mu\text{m}$ of the device. This is direct evidence that gas phase transportation of both gallium and nitrogen from the mask region to the device (i.e. partial pressure differences of both gallium or its compounds and nitrogen arising from the growth at the opening strip) acted as the driving force for mass transport mechanisms such as diffusion or forced gas flow to take place. Under such circumstances, the parasitic nucleation near the device region does not take place as the super-saturation was below the threshold whilst efficient lateral growth occurred. On the other hand, as parasitic growth developed away from the ELOG opening strip, the lateral growth rate is low and lateral growth almost stopped. Therefore, the gas phase diffusion length could be as long as more than $100\mu\text{m}$. Furthermore, an unexpected notch was formed during MOCVD growth at the Y-junction between the ELOG bar and contact pad, and the appearing facets must be slow-growth ones. It is believed that the smallest ratio of mass-flow rate to growth-interface length at the Y-junction promoted the transition from an angle of 120° to a concaved curved growth interface. This in turn leads to the appearance of both fast- and slow- growth facets forming at an angle of $\sim 90^\circ$ or larger. As growth continues, the fast-growth facets disappeared and a notch is formed. On the other hand, as these slow-growth facets at a notch have small absorption rates for both Ga and N, the surface diffusion rate on the top surface over a notch is reduced, and this led to enhanced vertical growth over a notch.

The impact of these results on the formation of ELOG cantilevers will be discussed.

Investigation of self-terminated growth of in-situ deposited SiN_x nano-mask

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The growth of GaN on foreign substrates like sapphire, silicon or SiC with a high lattice mismatch to GaN results in a high threading dislocation (TD) density, typically in the range of $10^9..10^{10}$ cm⁻². A common approach to reduce the TD density by approximately one order of magnitude is the in-situ deposition of a nano porous SiN_x mask on the GaN surface and its lateral overgrowth. However, the atomic structure of this nano-mask is not known so far, which is reflected by the common declaration SiN_x. Additionally, it is not completely understood why the nano-mask grows only as a (sub-) monolayer despite deposition times of several tens of seconds and why the nano-mask blocks further GaN growth on top of it.

In this work we present a detailed study on the atomic structure of the SiN_x nano-mask and its growth by a combination of aberration corrected high resolution transmission electron microscopy (HRTEM) and density functional theory (DFT) calculations. The samples were grown by metal-organic chemical vapor deposition (MOCVD) using standard growth conditions.

Our studies reveal a self-terminated, epitaxial (sub-) monolayer growth of the nano-mask on the underlying GaN. In cross-section HRTEM we find that the atomic columns of the mask-layer look identical in the [11-20] projection, while a periodicity of 3 atomic columns in the HRTEM pattern is visible along the mask-layer in the [1-100] projection. Such observation can be explained by a $\sqrt{3} \times \sqrt{3} R30^\circ$ surface reconstruction with different atomic species in the unit cell. In order to obtain chemical information about the mask-layer we have carried out STEM Z-contrast imaging and exit wave reconstruction. Based on our experimental results from both methods a number of possible structures have been developed, containing Si, Ga and V_{Ga}. DFT calculations were performed to obtain the relaxed structures of these configurations. Comparison of experimental and simulated images of the relaxed structures shows an excellent agreement for a complex structure including one Si, one Ga atom and one vacancy within the $\sqrt{3} \times \sqrt{3} R30^\circ$ unit cell. Such an atomic structure results in a SiGa₂N₃ composition for the nano-mask. Furthermore, DFT calculations show that the SiGa₂N₃ configuration is the thermodynamically most favorable one on the GaN (0001) surface and that a complete substitution of Ga by Si at the surface cannot be reached, even at the highest possible Si chemical potentials. The same holds for further growth of an additional SiN_x-layer on top of the first one, which explains the self-terminated growth of the mask.

Influence of reactor pressure on InGaN/GaN MQW growth and properties

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InGaN/GaN quantum wells are key element of all nitride optoelectronic devices. In spite of very good practical results obtained for III-N based LEDs and lasers, a correlation between growth regimes and device properties is not completely studied. Growth of InGaN layers by MOVPE is performed under very different conditions, and is very reactor sensitive, that makes difficult comparison results from different groups.

In general, reactor pressure influences indium incorporation in two differently directed ways: firstly, it is thermodynamically limited process (increased reactor pressure means increased pressure of indium vapor, and *higher* indium incorporation) [1]; secondly, parasitic reaction in a gas phase with particle formation [2] reduces indium incorporation as well as growth rate (increased reactor pressure means *lower* indium incorporation). Data published by different groups [3, 4, 5] covers different pressure regions and gives contradictory dependences of In incorporation with pressure.

The samples studied were grown on sapphire (0001) substrates using low-pressure MOVPE in a planetary reactor (AIX2000HT). The growth procedure was standard as described earlier [5]; active region contains several InGaN QWs with nominal thickness of 3 nm. Two series of samples were grown using reactor pressure variation from 100 to 940 mbar; these series differs by total gas flow during InGaN growth. Within the series only reactor pressure during InGaN deposition was varied. Structures were characterized by photo- and electroluminescence spectroscopy, X-ray diffractometry and transmission electron microscopy.

We observe that for lower total gas flow (19 slm) pressure increase from 100 to 900 mbar results in monothonical In incorporation decrease. For high total gas flow (49 slm) pressure increase from 100 to 400 mbar results in rise of indium incorporation (parasitic reactions in a gas phase suppressed); for pressures above 400 mbar parasitic reactions in a gas phase starts to dominate and In incorporation is decreased, forming a maximum at medium pressures. What is more interesting, it was revealed that In composition fluctuations increase with reactor pressure in both series, independently of the indium content variation, indicating changes in indium distribution within QWs.

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Influence of growth process scheme and parameters dispersion on the properties of AlGaN/GaN heterostructures

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Although MOVPE technique growth of nitrides is mature technology intensively developed by the last three decades, so far repeatability of the growth of device structures is strongly tool-dependent and methodology-dependent. Many equipment related factors like: reactor geometry, gas delivery system architecture and growth process parameters, type of precursors, III/V ratio, type of carrier gas and other factors influenced material quality and hence parameters of devices. Therefore, a simple description of the structures in a well known “sandwich” form without specifying the exact parameters of growth is ambiguous and does not allow to compare the results obtained on different epitaxial systems. This comes from the fact that particular structure can be grown in many different ways and only experiment recipe run on specific equipment can be sufficient description of the structure.

In this work authors will show how the growth process parameters can influence the device parameters for canonically the same “sandwich” structures. This will be presented on the examples of AlGaN/GaN heterostructures grown for HEMT's.

III/V ratio and growth speed (precursor injection speed) will be shown as a parameters which influences the properties of 2DEG. Also equipment related parameters, like reactor volume and gas delivery lines configuration and length will be presented as a strong factors of epitaxy at nanometer scale. These determinants in combination with growth kinetics of epitaxial layers (precursor incorporation and material decomposition and desorption) makes control of the growth and hence reproducibility difficult. Experimental work will be followed by mathematical calculations and simulations of growth process.

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Observation of Exciton-Phonon Interaction in AlGaN/AlN Quantum Wells

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Recent improvements in the epitaxial growth of AlN and AlGaN/AlN quantum wells (QWs) have opened windows of opportunity for studying their fundamental intrinsic properties. This includes the electron-phonon interaction which is governed by the motion of valence electrons in the crystal. As a result, phonons participate in the emission and absorption process, leading to enhanced absorption near the fundamental band edge and broadening of the emission peak. Among these interactions, the Fröhlich interaction, which is a Coulomb interaction between electrons (holes) and longitudinal electric field produced by the longitudinal-optical (LO) phonons, is the strongest one observed in III-nitride materials owing to its strong ionic nature [1]. The coupling of carriers with LO phonons influences the optical and transport properties in this material. In fact, exciton-phonon interaction has been observed in AlN showing LO replicas from free exciton [2] as well as in InGaN/GaN and GaN/AlGaN MQWs [3]. In this study, we report on the exciton-phonon interaction in AlGaN/AlN QW probed by photoluminescence (PL) spectroscopy.

AlGaN/AlN single QWs (~1 nm) were fabricated by modified migration enhanced epitaxy (MMEE) method [4]. An ArF (193 nm) laser was used for PL measurements. The PL spectrum acquired from Al_{0.87}Ga_{0.13}N/AlN SQW at 8.5 K showed broad linewidth (~200 meV) with features at the low energy side. The main emission line (5.708 eV) corresponds to exciton transition. To analyze the broad linewidth, fitting was performed using a Gaussian fitting function. The result has resolved four emission peaks with their peak energy positions equally separated by 107 meV, which is within the LO phonon energy of GaN (91 meV) and AlN (110 meV). These transition lines can be assigned to LO phonon replicas and an indicator of a strong exciton-phonon interaction in AlGaN/AlN QW. The relative intensities of the phonon replicas with the zero-phonon emission line were obtained using the Franck-Condon approximation [5]. The exciton-phonon coupling constant S , as determined by $I_n = I_0(S^n/n!)$, where I_0 and I_n are the emission intensity of the main emission line and n^{th} phonon replica, respectively, was estimated to be $S = 0.59$. This S is much higher compared to that of binary GaN ($S = 0.007$) and AlN ($S = 0.11$) [2], corroborating the strong exciton-phonon interaction in AlGaN/AlN QW. The influence of exciton localization with regard to the enhancement of exciton-phonon coupling is now under investigation.

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Optical anisotropy investigation from strained ($\text{In}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$) layers grown on (polar) c-sapphire

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Several researchers have intensively studied nonpolar or semipolar III-nitrides to reduce the polarization-induced electric field in nitride based LEDs. Therefore, this led researchers to investigate valence band mixing due to a small inclination of the substrates, which can change the polarization characteristics of III nitrides by investigating the optical polarisation properties.

We will present optical and structural studies of high quality $\text{In}_x\text{Ga}_{1-x}\text{N}$ thin film grown by MOCVD on (0001) sapphire (polar) with different InN composition ($0.03 < x < 0.24$) at different temperatures ($720 < T < 920$ °C). We studied the optical polarization by employing a polarizer in the photoluminescence (PL) setup. We used photoluminescence excitation (PLE) to investigate the bandgap of the films. X-ray diffraction (XRD), reciprocal space maps (RSM), Rutherford Backscattering spectrometry (RBS) and Raman spectroscopy were used to study the structural properties of the films. Surface morphology studies were carried out by Scanning Electron Microscope (SEM) and Atomic Force Microscope (AFM).

We observed that two $\text{In}_x\text{Ga}_{1-x}\text{N}$ films are fully strained and the sample with highest concentration (24%) was partially relaxed. X-ray diffraction (XRD) was used to determine the strain of the alloys using RSM while 2theta and omega scans were used to determine the InGaN peak position, quality of the samples and lattice parameter calculation which were compared. RSM showed that these InGaN layers are pseudomorphically except the one with 24% on GaN buffer layer. In addition, In contents were revealed by RSM measurements. Raman Spectroscopy using a UV excitation line (325 nm) showed the strained behavior of these samples. Raman spectroscopy showed that the strain of the sample grown with 15.8% In content is more than that grown with lower In content (9%).

We carried out PL and PLE measurements by using Xe lamp. The band gap of the samples was estimated from PLE measurements. Our preliminary optical polarization results showed an unexpected optical anisotropy from the fully strained $\text{In}_{15.8}\text{Ga}_{84.2}\text{N}$ film although it was grown on polar sapphire substrate. This optical anisotropy is similar to that of semi-polar and non-polar epilayers. In this work, we will discuss possible explanations for this phenomenon by comparing PL results with structural characteristics.

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Microphotoluminescence studies of phase separation in InGaN solid alloys

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In_xGa_{1-x}N solid alloys attract strong interest as potential material for developing light-emitting diodes (LED) and laser diodes (LD) of full visible range. This is because the energy gap of this ternary compound can be tuned between ~0.65 eV (currently accepted gap of InN) and ~3.4 eV (the bandgap of GaN). However, most of this range is covered by an alloy immiscibility region originating from the large difference between InN and GaN lattice constants, which causes phase separation phenomena during growth [1]. Typically, the phase separation manifests itself in the formation of In-rich clusters within an In-depleted matrix. This phenomenon has major effects on the device performance preventing fabrication of efficient green-to-red LDs and red LEDs.

In this work we present micro-photoluminescence (μ -PL) studies of the effect of phase separation in a 300 nm thick In_xGa_{1-x}N film grown by PAMBE on a 1- μ m-thick GaN(000 $\bar{1}$) buffer deposited on a c-Al₂O₃ substrate heated up to 640°C in accordance with IR pyrometer data obtained in a center area of the substrate. Due to the substrate temperature gradient (TG) of ~30°C/cm along certain direction the pronounced lateral gradient of the film composition is expected due to the strong dependence of the In incorporation rate on the growth temperature as it has been described in [2]. The average In-contents (x) were measured in several points of the film by electron probe microanalysis (EPMA) and X-ray diffraction mapping in the reciprocal space.

It was found that the average In content changes smoothly along the TG from 7% to 35%. Efficient phase separation at the applied growth temperatures is expected to occur in the In_xGa_{1-x}N films with x>0.1. μ -PL measurements reveal strong fluctuations of the emission spectrum at practically any average composition along the TG, emerging on the scale of approximately 1 μ m that is close to the spatial resolution of the μ -PL setup. This PL inhomogeneity reflects the respective inhomogeneous distribution of In occurring on the same size-scale. Surprisingly, the μ -PL spectra measured at varies points with different average composition show almost identical emission bands with fixed wavelengths: 495-505 nm (blue band), 510-525 nm (green band), 565-585 nm (orange band), and 590-650 nm (red band). This behavior implies emergence of at least four phases with fixed In content, which are almost independent of the average film composition and the variation of the growth temperature. The change of the average composition results mainly in the modification of the relative volumes filled by In_xGa_{1-x}N with various fixed compositions. These observations can hardly be explained in the framework of the classical description of the phenomenon of spinodal decomposition [1] and implies important role of other factors such as surface morphology (grain size), adatoms kinetics, distribution of threading dislocations, local stress in the separated alloys etc. These results show possibility of further improvements of the luminescence properties of InGaN structures with the relatively high In content by growing films having optimum average compositions.

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Cathodoluminescence study of InGaN quantum well grown on GaN annular structure by metal-organic chemical vapor deposition

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(In, Ga)N-based III-nitride semiconductor materials have been viewed as the most promising materials for the applications of blue and green light emitting devices such as light-emitting diodes (LEDs) and laser diodes. Although the InGaN alloy can have wide range of visible wavelength by changing the In composition, it is very hard to grow high quality epilayers of InGaN because of the thermal instability as well as the large lattice and thermal mismatches. InGaN layer was commonly grown on *c*-plane GaN by metal-organic chemical vapor deposition (MOCVD). Large spontaneous and piezoelectric polarization of InGaN on *c*-plane GaN leads to poor wave-function overlap resulting in poor emission efficiency. To reduce the polarization, it is introduced to grow semi-polar and non-polar facet of GaN. Although there were lots of research to grow semi-polar facets such as {10-11} and {11-22}, it is lack of study on the optical property between many kinds of semi-polar facets. Especially, optical properties of {10-11} and {11-22} semi-polar facets were still ambiguous despite their similar structural properties. In this study, by utilizing InGaN multi-quantum well on GaN annular structure, we have investigated comparative study on the optical properties of semi-polar facets between {10-11} and {11-22}.

In order to characterize optical properties of these semi-polar facets, {10-11} and {11-22} facets have to be formed with same growth condition. When GaN is grown on SiO₂ mask with ring pattern, the GaN annular structure is formed and the annular structure includes {0001}, {10-11} and {11-22} facets followed by 5 period of InGaN/GaN multi-quantum well structure. Figure 1 shows top-view scanning electron microscope (SEM) image of an InGaN/GaN annular structure and cathodoluminescence (CL) spectra measured when the electron beam excite the position p1, and p2 at 300 K. While outside sidewalls of annular structure are {10-11}, inside sidewalls have both {10-11} and {11-22} facets. InGaN emission from {10-11} shows red-shift emission with broad spectrum compared to emission from {11-22}. We also measured monochromatic CL mapping images, as shown in Figure 2. We confirmed that {11-22} facets show shorter wavelength emission centered at 420 nm, while {10-11} facets have longer wavelength emission centered at 470 nm. To characterized optical property, we also carried out micro-photoluminescence (PL) and PL excitation at 10 K. The GaN annular structure is good structure to understand detailed optical property of semi-polar facets and this study will provide fruitful information for growth of semi-polar GaN structure.

Growth Mechanisms of Catalyst-free and Mask-free Heteroepitaxial GaN Nanorods and Microrods on Si (111) and Si (100) Substrates Grown by MOVPE

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Heteroepitaxial GaN nano- and micro-rods (NMRs) are one of the most promising structures for high performance optoelectronic devices such as light emitting diodes [1], lasers [2], solar cells [3] integrated with Si-based electric circuits due to their low dislocation density and high surface to volume ratio. However, heteroepitaxial GaN NMRs growth using a metal-organic vapor phase epitaxy (MOVPE) machine is not easy due to their long surface diffusion length at high growth temperature of MOVPE above 1000 °C. Recently some research groups reported the fabrication of the heteroepitaxial GaN NMRs by using MOVPE with vapor-liquid-solid (VLS) technique assisted by metal catalyst [4-5]. However, in the case of the VLS technique, metal catalysts may act as impurities, and the GaN NMRs produced in this method have poor directionality.

We have successfully grown the vertically well aligned GaN NMRs on Si (111) substrate and the 45 degree-tilted GaN NMRs on Si (100) by means of self-catalytic growth methods with pulsed-flow injection of precursors. To grow the GaN NMRs with high aspect ratio, we varied the growth conditions such as the growth temperature, reactor pressure, and V/III molar ratio. We confirmed that the surface morphology of GaN was strongly influenced by the surface diffusion of Ga and N adatoms related to the surrounding environment during growth, and we carried out theoretical studies about the relation between the reactor pressure and the growth rate of NMRs. Using the optimized growth condition, we investigated the effect of biaxial strain on GaN NMRs with the various substrates, which induced the different strains on the GaN NMRs. The density functional theory was employed to calculate the surface energy varied by the biaxial strains on NMRs. From these results, we successfully explained the growth mechanism of catalyst-free and mask-free heteroepitaxial GaN NMRs on Si (111) substrate. Detailed experimental results will be discussed.

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Lattice dynamics of short period GaN/AlN superlattices. Theory and experiment

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We report the results of theoretical and experimental studies of lattice dynamics of short-period GaN/AlN SLs which have a great potentiality for a wide range of electronic and photonic devices. *Ab initio* calculations of the phonon spectrum of SLs were carried out with the ABINIT package using a plane-wave pseudopotential approach to the density functional theory. The calculations were performed in the local density approximation with the Fritz-Haber pseudopotentials in which the semicore Ga 3d electrons are treated as valence electrons. The procedure involved several stages: optimization of the geometry of the structure, the calculation of the ground state, the calculation of the phonon spectrum and mode eigenvectors, and the calculation of the Raman susceptibility tensor. To study the effect of the SL period on the phonon spectrum, three types of (GaN)_n/(AlN)_n SLs ($n = 2, 4, 6$) were tested. Here n is the number of GaN (and AlN) monolayers per SL unit cell. The frequencies of all phonon modes were calculated and the mode displacement patterns have been established. The number and symmetry of normal modes in the calculated spectrum agree with the results of group-symmetry analysis. Analysis of eigenvectors of the phonon modes led to the conclusion that there are two types of phonon modes in the phonon spectrum of short-period GaN/AlN SLs. The modes of first type are the propagating modes, which involve the in-phase displacements of the cations or anions both in GaN and AlN layers. The modes of the second type are the confined modes. These modes involve the displacements of atoms only in GaN (or AlN) layers, while the atoms of the other layers AlN (or GaN) almost do not move. It is shown that the confinement of phonon modes is valid even for SL with the thinnest layers ($n = 2$). The propagating modes reflect the averaged characteristics of the SL as a whole and the confined modes give information about the individual characteristics of each layer. Our finding of the two types of modes could be the base for the development of quantitative diagnostics of such structures. On the basis of the Raman susceptibility tensor, the theoretical Raman spectra were calculated and compared with experimental ones. The experimental Raman spectra were obtained for GaN/AlN SLs grown by MOVPE. The samples were grown in a horizontal flow reactor at temperature of 1050° C on (0001) Al₂O₃ using GaN and AlN buffer layers [1]. The period of SLs varied from 2 to 6 nm, and the thickness of the structures ranged from 0.3 to 1 μm. The results of *ab initio* calculations of frequencies and intensities of Raman modes agree well with the experimental data. The combination of theory and experiment allowed us to develop a new optical method of quantitative evaluation of such important parameter of short-period GaN/AlN SLs as the strain in the individual layers forming the structure. In addition, it was demonstrated for the first time that the thicknesses of layers of short-period GaN/AlN SLs can be estimated with the accuracy up to a monolayer by Raman spectroscopy.

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Polarization effects in light emission from strained AlGaN-based heterostructures

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State-of-the-art AlGaN-based UV LEDs require further improvement of their efficiency that is, in part, limited by the polarization of emitted light. Recent experiments have demonstrated a rise in the TM-polarized component of light, while the AlN fraction in the AlGaN alloys is increased. In particular, this resulted in the polarization switching upon the emission wavelength variation observed in both bulk AlGaN [1] and thin AlGaN-based quantum wells [2,3]. The nature of the effect was found to originate from the non-ordinary structure of the AlGaN valence band, namely from the character of the topmost hole state depending on the AlN fraction in the alloys and switching from the heavy-hole to the split-off-hole ones. The polarization changes were accompanied by transformation of the emission pattern from nearly isotropic to that dominating in the crystal C-plane. The latter is believed to influence the light extraction efficiency from a UV LED die and, hence, its total emission efficiency. Being well understood qualitatively, the above polarization effects have not yet been described satisfactory on the quantitative level.

This theoretical study is aimed at analyzing the polarization phenomena in the light emission from strained Al(In)GaN alloys with account of all the key factors determining the polarization degree: the strain effects on the semiconductor band structure, the strong non-parabolicity of the valence subbands producing complex density of states for holes of particular types, the competitive populations of the hole subbands, and the wave-vector dispersion of the optical matrix elements. For this purpose, we considered strained Al(In)GaN layers, i. e. those having a small amount of indium, grown on C-plane buffer layers that control the in-plane lattice constants of the materials. The model based on the 6×6 Bir-Pikus Hamiltonian included elastic strain to predict the energy spectra of heavy, light, and split-off holes. These spectra were then used for numerical calculating the densities of states for each type of holes and their approximating by appropriate analytical expressions. The hole densities of states and the wave-vector dependent optical matrix elements were finally employed to simulate the emission pattern, spectrum, and polarization degree of light emitted from an Al(In)GaN alloy.

The polarization degree of light emission from a strained Al(In)GaN alloy is found to depend remarkably on the observation angle. In particular, in the case of in-plane light emission, our simulations demonstrate good agreement with the data of [1-3], providing the polarization switching at the wavelength of ~300 nm. Strongly anisotropic emission pattern predicted for Al(In)GaN alloys is found to depend on competition of the TE- and TM-polarized photons, generation of which occurs with their own emission patterns primarily dependent on the alloy composition.

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Time-resolved photoluminescence studies on HVPE freestanding GaN substrates exhibiting record-long positron diffusion length

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As a solution to concerns about energy crisis, exploitation of Al_xGa_{1-x}N / GaN power transistors and InGaN quantum well light-emitting diodes (LEDs) is one of the significant ways for drastically decreasing total energy consumption [1]. Although commercially available blue, green, and white InGaN LEDs fabricated on significantly defective GaN having the threading dislocations (TDs) as high as 10⁹ cm⁻² exhibit reasonably bright emissions with the aid of exciton localization, fabrication of low TD density, high-purity, and large-area polar freestanding GaN (FS-GaN) wafers of polar, nonpolar, and semipolar planes is preferred to achieve high-performance and reliable devices.

Approximately 1-cm-thick, transparent *c*-plane GaN boules were grown using a vertical-flow halide vapor phase epitaxy (HVPE) apparatus [2]. Appropriate amount of gaseous HCl was flowed on heated Ga, and NH₃ was supplied from a separate gas line. Typical growth temperature and pressure were 1050 °C and atmospheric pressure, respectively. The growth duration was varied from 55 to 96 hrs. Approximately 325-μm-thick, 10 × 10-mm²-area, *c*-plane and *m*-plane FS-GaN wafers were sliced [2,3] from GaN boules grown under various conditions.

As time-resolved photoluminescence (PL) signals for most of the samples exhibit biexponential decay shape, the fitted fast component (τ_1) that dominates the overall PL intensity is used to compare various FS-GaN samples [4]. Obviously, τ_1 increases with decreasing *S* parameter (size or concentration of V_{III}). Because *S* of the present samples has already reached characteristic value for vacancy-free GaN (S_{free}), τ_1 distributes from 0.1 ns to 1.1 ns for the same *S* ($=S_{free}$). On the other hand, τ_1 increases with the increase in positron diffusion length (L_+), and eventually reaches 1.1 ns for $L_+=116$ nm. These results confirm that fundamental limiting factor for the nonradiative lifetime and thereby internal quantum efficiency at room temperature is gross concentration of point defects and complexes, which are incorporated with Ga vacancies (V_{Ga}) [4]. Certain defects, namely V_{Ga} -X defect complexes, are the most probable candidate of nonradiative recombination centers [4-6].

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Full molar fractional Raman spectra from RF-MBE grown $\text{In}_x\text{Al}_{1-x}\text{N}$ alloys ($0 < x < 1$)

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$\text{In}_x\text{Al}_{1-x}\text{N}$ alloys system is one of hopeful semiconductor one for various device applications such as solar cell, because it is the direct transitional and the wide band-gaps ranging system from 0.64 eV for InN to 6.2 eV for AlN. However, it is a rather difficult to obtain the $\text{In}_x\text{Al}_{1-x}\text{N}$ ($0 < x < 1$) alloys with device quality, because of the large different natures of the mother materials of InN and AlN. Therefore, there are very few reports on the systematic research of the Raman spectra from the full molar fractional range of the $\text{In}_x\text{Al}_{1-x}\text{N}$ alloys, in order to investigate the optimized growth conditions by the Raman mode observations in the c-plane back-scattering geometry. In this paper, we reported about the preliminary results for the systematic investigation of the Raman spectra from RF-MBE grown $\text{In}_x\text{Al}_{1-x}\text{N}$ alloys with the full molar fraction range.

The $\text{In}_x\text{Al}_{1-x}\text{N}$ alloys were grown on sapphire (0001) substrates by RF-MBE, in which the 7N metals of In and Al and the radio frequency excited N_2 plasma were used as the precursors. The RF power was fixed at 330 W, and the beam flux of In and Al were varied from 6.6×10^{-8} Torr to 4.0×10^{-7} Torr and from 1.0×10^{-7} Torr to 2.4×10^{-7} Torr, respectively. The growth temperature was varied from 600 to 650 °C. Both the In/(In + Al) beam flux ratio and the growth temperature were varied to control the InN molar fractions. The Raman observations were performed in the c-plane back-scattering geometry at room temperature with the excitation wavelength of 532nm. The InN molar fractions were determined by the X-ray diffraction (XRD) method.

We observed the rather broad Raman peaks which can be decomposed three Raman modes of the E_2 (high), A_1 (LO) and the forbidden B_1 (high) modes fitted with the theoretical results [1, 2]. We can also observe that the E_2 (high) mode in the $\text{In}_x\text{Al}_{1-x}\text{N}$ alloys showed the two-mode behavior of the InN-like and the AlN-like E_2 (high) modes. The results strongly indicate that the Raman spectroscopy is very useful for the investigation of the structural properties of the $\text{In}_x\text{Al}_{1-x}\text{N}$ alloys to obtain the optimum growth conditions.

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Unstable luminescence of nitrides under electron-beam irradiation

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In spite of successful commercialization of nitride LEDs there are still a number of unsolved problems. One of such problems is the fast degradation of LEDs. It is known that nitrides could be degraded by ultraviolet and electron injections [1]. Therefore study of luminescence stability under electron beam irradiation is strongly important for understanding of the degradation mechanism in nitrides.

Unstable behavior of luminescence of nitrides was observed formerly [2-4]. Several mechanisms were supposed to be responsible for this effect. It was assumed that an enhanced diffusion of nonradiative recombination centers or impurities can be induced by electron beam irradiation [2]. In other studies it was proposed that a formation of metastable states and trapping of electrons took place [3]. However detailed study of this effect was not made yet.

In this work an analysis of the unstable luminescence in nitrides was made. Samples grown by different techniques (PAMBE, MOCVD, HVPE) and with different kind of doping were studied. The experiments were made at Camebax electron microprobe. Cathodoluminescent (CL) measurements were taken using original CL spectrometer [5]. It was found that unstable behavior is typical both for "band-to-band" transitions and for defect luminescence. Under several conditions only luminescence decay with typical times of dozens and hundreds of seconds was observed. On other samples or/and under other conditions more complicated nonmonotonic behavior of luminescence took place. Moreover the effect of the luminescence time-instability was revealed in nitride heterostructures as well. We found out that the luminescence of quantum wells or barrier layers has a complicated time-dependence. In several samples electron-beam irradiation resulted in irreversible changes of spectral position and intensity of luminescence spectra.

Our results allow to conclude that the effect studied is not connected with microvolume overheat as unstable luminescence was observed in different range of temperatures (77 K-300 K). The dependence of the effect on the electron beam current is very strong. It was found out that there is a threshold valuation of the electron beam current for rise processes. It was shown that during the electron beam irradiation (~400 sec) electric charge ($\sim 10^{-8}$ C÷ 10^{-10} C) is trapped in the samples. The influence of the surfaces and interfaces (in heterostructures) on the effect studied was proposed. The nature of the electron traps is discussed.

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Temperature-resolved photoluminescence of nonpolar InGaN/GaN multiple quantum well heterostructures grown on LiAlO₂

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Gallium nitride and related compounds grown in nonpolar directions attract much attention due to a number of unique properties such as the ability to emit and detect polarized light and the absence of built-in electric fields along the growth direction suppressing the quantum confined Stark effect. In the present work, we investigated temperature-dependent photoluminescence (PL) of a series of m-plane In_xGa_{1-x}N/GaN multiple quantum well (MQW) heterostructures grown on LiAlO₂ substrates with indium contents x being in the range from 5% to 30%. The maximal integral PL intensity was measured for the sample with $x=10\%$ which is due to a competition between improved localization of photogenerated carriers and strain-induced nonradiative defect generation in InGaN with larger In content. Clear S-shaped temperature dependences of PL band positions were observed for all samples at low excitation level (~ 1 W/cm²). The “dip” in the temperature dependence of the PL spectral position shifted to higher temperatures with rising In content x . This is caused by localization of nonequilibrium charge carriers at deeper localized states in InGaN with higher indium content. The S-shaped behavior vanished at higher excitation level (~ 300 kW/cm²) due to a saturation of deep localized states by photogenerated carriers. Temperature-induced PL quenching was weaker at high excitation levels; this can be attributed to a decreasing impact of nonradiative centers at elevated excitation levels. Temperature quenching was more pronounced for samples with lower x at low excitation levels indicating a smaller activation energy of nonradiative channels in InGaN with lower indium contents (i. e. a weaker localization of photogenerated carriers). The observed pronounced S-shaped behavior of the PL band position with temperature indicates a substantial inhomogeneity of indium distribution even for InGaN with low x (5%) and is rather unexpected. This can be caused by the inhomogeneous in-plane lattice strain in the GaN buffer layer grown on LiAlO₂. The strain modulation in the GaN buffer layer can cause, in turn, an inhomogeneity of indium distribution in InGaN layers. The degree of polarization (DOP) of PL emission from the InGaN layers was also measured as a function of temperature for samples with different indium contents. The energy difference ΔE between the two topmost valence subbands in InGaN was evaluated from this result using the Fermi statistics distribution. The energy difference rises approximately from 95 meV to 150 meV with an indium content increase from 5% to 30%. The result is well consistent with data from [1]. The high values of ΔE can be explained by strain in the GaN buffer layers on the LiAlO₂ substrate. At low temperature, an irregular long-wavelength shift of the less intense $E\parallel c$ -polarized PL component is observed which is inconsistent with the InGaN band structure. The probable reason for the phenomenon is a lower DOP of PL from deeper *In*-clusters because of filling of states corresponding to the second valence subband [2].

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The effect of the magnetic field on the micro-photoluminescence spectra of the InGaN/GaN MQW structures doped with Eu, Sm and Eu+Sm

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The effect of the magnetic field strength and excitation power on the micro-photoluminescence (μ -PL) spectra of the undoped and doped with Eu, Sm and Eu+Sm InGaN/GaN MQW structures has been investigated. Diffusion technique was used for doping with these impurities. Magnetic field in Faraday geometry in the range of 0 – 5 T was provided by superconductive solenoid. The upper limit of impurity concentrations as was determined by x-ray fluorescence technique was around $5 \times 10^{18} \text{ cm}^{-3}$. For the determination of the charge states of Eu and Sm Messbauer spectroscopy was used.

μ -PL spectra of undoped InGaN/GaN structures showed the decrease of their PL intensity with increase of magnetic field strength at high optical excitation level, while spectral position of the PL band maxima (λ_{max}) remained unchanged. In the Eu-doped InGaN/GaN structures under the same conditions as in undoped structures the greater changes compare to those in undoped structures were observed: considerable decrease of the PL intensity in the short range of μ -PL spectrum while only minor changes were recorded in the long wave range region.

It is known that Sm as well as Eu is Van-Vleck paramagnetic impurity. The effects of Sm impurity on the μ -PL spectrum of InGaN/GaN structures are differed compare to those of Eu under the same conditions: PL intensity increases with increase of magnetic field which is accompanied by the blue-shift of λ_{max} by approximately 0.8 nm. Increase of both the magnetic field strength and excitation intensity (by one order of magnitude) results in the appearance of two PL bands in short-wave region of PL spectra ($\lambda = 425 \text{ nm}$ and 430 nm). They are of equal PL intensity which decreases with increasing of the magnetic field strength.

Simultaneous doping with Eu and Sm leads to the changes in the short-range ($434 \text{ nm} < \lambda < 460 \text{ nm}$) of the PL spectra: three PL bands of different intensity appear. Also several PL bands of smaller intensity are recorded in the long-wave region ($735 \text{ nm} < \lambda < 800 \text{ nm}$) of PL spectra. The increase of the strength of magnetic field leads to only minor decrease of PL intensity in the short-wave range region, leaving both spectral position and PL intensity of PL lines in the long-wave region unchanged.

According to the data of Messbauer spectroscopy the main charge state in InGaN/GaN structures doped with Eu is Eu^{3+} though smaller concentration of Eu^{2+} is also present. The latter together with the residual impurity of Fe atoms could be ferromagnetic impurity. As to Sm, only charge state 3+ (absence of 2+ state) was revealed in Messbauer spectra. This circumstance is the suggested reason to observe essentially different effects of doping of the studied InGaN/GaN structures with Eu and Sm on their μ -PL spectra.

Cubic III-nitride coupled quantum wells towards unipolar optically pumped lasers

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Today III-nitrides are the material of choice for manifold device applications like light emitting diodes, laser diodes or field-effect transistors. Due to the large band discontinuity between AlN and GaN, novel nitride-devices based on intersubband transitions, like quantum well infrared photodetectors, quantum cascade lasers (QCLs) or optically pumped lasers operating at telecommunication wavelengths are proposed. In state of the art devices wurtzite nitrides are used. Due to the hexagonal symmetry strong intrinsic piezoelectric and pyroelectric fields are present at the hetero-interfaces. These built in fields reduce transition probability in quantum wells (QWs) and are therefore undesirable in optical devices. In multi-quantum-wells or superlattices these internal fields complicate the design and limit the tunability of intersubband transition energies. Hence the growth of non-polar and semi-polar nitrides has found increasing interest in the last years to avoid these strong internal fields. However, the electrical, optical and structural properties show strong lateral anisotropy since the polarization field is now in the plane of growth. An alternative way to fabricate group III-nitrides without spontaneous polarization fields is the growth of meta-stable non-polar cubic group III-nitrides [1]. During the last years, a lot of preliminary work towards unipolar optoelectronic devices like QCL or optically pumped lasers based on cubic nitrides has been achieved. The large band-offset between c-GaN and c-AlN has been determined [2]. Intersubband absorption in the near-infrared to terahertz spectral range was measured [3] and resonant tunnelling through c-AlN double barriers was shown [4] which forms a base for future optically pumped lasers based on cubic group III-nitrides. Optical and structural properties of asymmetric coupled c-GaN/c-AlN quantum wells are studied. The samples consist of a 50 nm c-GaN buffer grown on 3C-SiC. The active region is formed by a 3 nm c-AlN barrier, a 2.1 nm silicon doped c-GaN QW and a 1.9-1.6 nm undoped c-GaN QW separated by a 0.9 nm c-AlN barrier. This structure is repeated up to 100 times. Phase purity and partial relaxation of the superlattice is observed in reciprocal space maps measured by high resolution x-ray diffraction. Optical properties of our samples are investigated using cathodoluminescence spectroscopy. A clear shift in the emission energy associated with the thickness of the undoped QW can be observed. Furthermore clear TM-polarized infrared absorption is measured using FTIR. Measured transition energies are compared to model calculations using a Schrödinger-Poisson solver based on an effective mass model (nextnano³).

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Confocal micro-Raman spectroscopy in-depth and across the cleaved edge of vertically designed GaN Gunn-diode structures

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Gallium nitride based semiconductor structures are promising for fabrication of high-frequency Gunn-diodes with vertical device design which may achieve THz operation regime. Heteroepitaxial GaN films grown by molecular-beam epitaxy are known to contain high concentration of defects and have a predominantly mosaic structure consisting of c-oriented nanocolumns, which affects the breakdown voltage, the carrier mobility and device reliability. For optimization of technology of high quality GaN heterostructures, new nondestructive methods are required for evaluating the deformations, crystal structure quality, and free carrier concentration within nitride epitaxial films with a high spatial resolution. Confocal Raman micro-spectroscopy allows non-destructive evaluation of structural and electronic properties of GaN diode structures with submicron spatial resolution.

GaN Gunn-diode structures ($n^{++}/n_0/n^{++}$) were grown on Al₂O₃ (0001) substrates by plasma-assisted molecular beam epitaxy at temperature of 800°C. The structure was as follows: a 4 μm thick Si-doped n^{++} -GaN back contact layer; 2 μm thick undoped n_0 -GaN channel layer, and a 0.5 μm thick Si-doped n^{++} -GaN top contact layer. The nominal Si doping concentration of the n^{++} -GaN layers was $\sim 2.7 \times 10^{18} \text{ cm}^{-3}$. Micro-Raman measurements were performed using JY T64000 triple spectrometer equipped with an Olympus BX41 confocal optical microscope and piezo-driven XYZ scanning stage. The 488.0 nm line of Ar⁺/Kr⁺ laser was used for excitation. Depth and cleaved edge profiling polarized Raman measurements were performed in $z(y,y)-z$ and $x(y,y)-x$ geometries with a spatial step of 0.1 μm.

Spatial profiles of the $E_2(\text{high})$ -phonon mode and the coupled ω^- - plasmon-LO-phonon mode frequencies and intensities correlate well with the design of the investigated Gunn-diode structure and reveal hidden features of the residual strain in the device. Gradual decrease in frequency of the $E_2(\text{high})$ -phonon mode is observed when scanning spatially from the Al₂O₃ substrate to structure's surface. In addition, profiling near a cleaved edge revealed a near complete relaxation of the strain due to the free surface. Further study demonstrated a gradual decrease in strain beginning at a distance of $\sim 50 \mu\text{m}$ from the cleaved edge.

The frequency profiles of the ω^- mode (concentration of free carriers) demonstrated significant contrast through the undoped n_0 -GaN region validating the expected reduction in free carriers. However, profiles near the structure's cleaved edge revealed an unexpected reduction in carrier concentration near the free surface. This is believed to be due to the existence of structural defects in the region near the cleaved edge of the structure.

Thus, scanning confocal micro-Raman spectroscopy is shown to be an effective tool for studying the spatial distribution of strains, crystal quality, and free carrier concentration in GaN-based devices with submicron resolution.

Confocal Raman and photoluminescence microscopy of light-emitting heterostructures with multiple InGaN/GaN quantum wells

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The development of blue light emitting diodes based on InGaN/GaN multilayer heterostructures continues to be of considerable technological interest. Device performance depends on spatial distribution of structural defects, resulting from clustering in InGaN-alloys phase-segregation, dislocation boundaries of domains. Scanning confocal Raman spectroscopy is a nondestructive method for evaluating the degree of spatial homogeneity of the crystal structure of InGaN/GaN heterostructures with submicron spatial resolution, which allows to understand the correlation between their structural and light-emitting properties. In this work, we present Raman and photoluminescence (PL) spectroscopic studies on the influence of parameters of active InGaN/GaN superlattice on their phonon and radiative properties.

Investigated multilayer InGaN/GaN heterostructures were grown by MOCVD on (0001)-oriented sapphire substrates with predeposited low-temperature GaN template layer with thickness of 30 nm. Light-emitting structures consisted of n-GaN:Si layer with thickness of 3.5 μm ($N_d \approx 5 \times 10^{18} \text{ cm}^{-3}$), 5-period buffer $\text{In}_{0,08}\text{Ga}_{0,92}\text{N}$ (2.5 nm)/GaN(4 nm) superlattice, active InGaN/GaN region, and top p^+ -GaN:Mg layer with thickness of $\sim 0.1 \mu\text{m}$ ($N_a \approx 10^{20} \text{ cm}^{-3}$). Series of investigated samples had active regions (superlattices) containing 1, 3 and 5 $\text{In}_{0,20}\text{Ga}_{0,80}\text{N}$ quantum wells with thickness of 2,5 nm, and separated by 9 nm thick GaN barriers.

The radiative recombination of nonequilibrium charge carriers in multilayer InGaN/GaN structures was investigated by scanning along the growth direction of the structure ($E_{\text{exc}} = 3.81 \text{ eV}$). Obtained non-uniform profiles of depth distribution of the intensity, energy position and half-width of the exciton emission band for GaN buffer layer ($\approx 3,4 \text{ eV}$), InGaN quantum wells of the barrier superlattice ($\approx 3,06 \text{ eV}$, $\Gamma \approx 0,13 \text{ eV}$) and the active region ($\approx 2,66 \text{ eV}$, $\Gamma \approx 0,12 \text{ eV}$) which are determined by local structural and compositional disorder and non-uniform elastic strains in nitride layers. Based on the energy position of InGaN quantum wells PL bands (and taking into account the contribution of elastic strains obtained from the Raman spectra) the changes in the composition in depth of the structures were analyzed.

Analysis of frequency position and half-width of $E_2(\text{high})$ phonon band of $\text{In}_x\text{Ga}_{1-x}\text{N}$ in the depth Raman spectra made it possible to obtain a direct evidence of the gradient asymmetric spatial profile of the relaxation of elastic strains in the nitride layers along the growth direction of the InGaN/GaN structure. Strains in InGaN-layers change in the range of $(0.9\div 1.04) \times 10^{-2}$. Also, the resonant Raman spectra of InGaN/GaN structures in $x(\text{zz})-x$ geometry were measured at excitation with energies from 1.83 to 3.81 eV, which is close to the real electronic transitions of InGaN quantum wells. Resonance Raman scattering on quasi-LO phonons of buffer superlattice ($\approx 729 \text{ cm}^{-1}$) and active region ($\approx 744 \text{ cm}^{-1}$) of InGaN/GaN structure was observed. Registration in $x(\text{zz})-x$ Raman spectra of forbidden by the selection rules scattering on quasi-LO phonons can be caused by a large amount of structural defects in the InGaN quantum wells.

Obtained results demonstrate the efficiency of confocal Raman and PL studies of the structural and radiative inhomogeneities of InGaN/GaN structures at submicron level.

Peculiarities of photoluminescence efficiency dependence on excitation intensity in GaN/Al₂O₃ epilayers

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GaN layers grown on Al₂O₃ were investigated by photoluminescence (PL) and light induced grating technique (LITG) at various excitation levels. The excitation-dependent measurements were carried out using a pulsed N₂ laser with the optical power density I_{exc} in the range from 1.5 to 4600 kW/cm². The carrier recombination and diffusion processes were monitored by LITG under excitation by the 3-rd harmonic of an Nd:YAG laser. The carrier lifetime τ_R and ambipolar diffusion coefficient D were determined by varying the period of the induced dynamic diffraction grating [1].

All the investigated samples showed increasing PL efficiency of the near band edge (NBE) peak with increasing excitation level due to the increasing of the contribution of bimolecular radiative recombination (Bn^2) to the recombination rate. As a rule, the NBE PL had a higher efficiency for higher carrier lifetime values at high excitation and for lower carrier lifetime values at low excitation, respectively. The samples with higher background electron density n_e showed higher PL efficiency at low excitation, whereas at elevated excitation PL became less intensive. The observed crossing of the excitation dependences of the PL efficiency of different GaN/Al₂O₃ samples can be explained by the different concentration of defects acting as donors. At low excitation, the higher n_e promotes increasing the radiative term $B(n+n_e)n$ whereas at high excitation (corresponding to high n) the contribution of n_e decreases. As a rule, an increased dislocation density leads to an increase of n_e resulting in the crossing of the excitation dependences of PL efficiency. The samples with higher dislocation density have higher PL efficiency at low excitation. Therefore, only PL efficiency measured at a high excitation level can give reliable information about the quality of a GaN layer.

The NBE PL efficiency increases with excitation, but the drop at a very high excitation level (>2500 kW/cm²) is observed while collecting PL emission from surface. To check a reason of this drop, we measured PL detected from the edge of the sample. The drop of PL efficiency detected from surface was found to be accompanied by a sharp increase of PL efficiency detected from edge. The PL spectra measured from edge have a pronounced narrow peak at ~ 371 nm with a very sharp dependence on excitation that can be ascribed to electron-hole plasma related stimulated emission propagating along the in-plane direction [2]. Therefore, the reason of the observed efficiency drop of PL measured from surface at the high excitation level is the saturation of the density of photogenerated carriers caused by stimulated recombination.

We estimated the spatial distribution of carrier concentration with the measured τ_R and D values taken into account. Based on this data, a possible contribution of Auger recombination to the observed PL efficiency drop is discussed.

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Dramatic thermal quenching of photoluminescence in Zn-doped GaN

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Variation of temperature is widely used in photoluminescence (PL) studies to explain effects related to point defects in semiconductors and to determine some of their important properties. In conductive *n*-type semiconductors, the behavior of the defect-related PL with changing temperature is relatively well understood. For example, in *n*-type GaN, most of the PL bands are related to different types of acceptors. These PL bands are quenched above a characteristic temperature T_0 . When the logarithm of the PL intensity is plotted as a function of inverse temperature (the Arrhenius plot), the slope of this dependence at $T > T_0$ reveals the acceptor's ionization energy. Both T_0 and the slope of the thermal quenching increase linearly with increasing the ionization energy of the acceptor. However, for high-resistivity semiconductors, the behavior of PL is studied much less and is not well understood.

We report on the dramatic thermal quenching of the blue luminescence (BL) band in high-resistivity Zn-doped GaN. The BL band from undoped and Zn-doped GaN has a maximum at 2.9 eV, a zero-phonon line at 3.10 eV and a characteristic fine structure at low temperatures. In conductive *n*-type GaN samples, the thermal quenching of this band begins at $T_0 = 200$ K, independent of excitation intensity. The activation energy of this thermal quenching is about 0.35 eV, in agreement with the ionization energy of the Zn_{Ga} acceptor. However, in high-resistivity Zn-doped GaN, the BL intensity drops by several orders of magnitude at a characteristic temperature T^* , and this temperature increases with increasing excitation intensity; *i.e.*, the quenching is tunable. For example, in one sample, T^* increased from 130 to 270 K with increasing excitation intensity by six orders of magnitude. When the temperature of the sample is fixed, the dependence of PL intensity on the excitation intensity exhibits an abrupt, stepwise increase at a certain excitation intensity, and the value of the latter depends on temperature.

This unusual PL behavior was reproduced in a large number of Zn-doped GaN samples. All the effects can be explained by a model of a high-resistivity semiconductor with three types of defects: a shallow donor, the Zn_{Ga} acceptor, and a deep donor. The latter is responsible for the nonradiative recombination. The abrupt and tunable thermal quenching of the Zn-related BL band in high-resistivity GaN samples is caused by a sudden transition of the system from the inverse population of levels at $T < T^*$ (where low-resistivity *n*-type conductivity under illumination is predicted) to the quasi-equilibrium population at $T > T^*$ (a *p*-type, or high-resistivity *n*-type conductivity). This transition is initiated by the thermal emission of holes from the Zn_{Ga} acceptor to the valence band and their subsequent recombination with electrons via deep nonradiative defects. Although only few reports on the tunable thermal quenching in semiconductors could be found, we predict that this phenomenon may be quite common in high-quality samples. In particular, a very abrupt quenching (a drop in PL intensity by several orders of magnitude in the temperature range of about 10 K) is expected in high-resistivity wide-bandgap semiconductors containing relatively deep acceptors and nonradiative defects with certain parameters. The origin of the nonradiative defects in GaN will be discussed.

Electrical and optical properties of bulk GaN substrates studied by Kelvin probe and photoluminescence

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Epitaxy of GaN films on bulk GaN substrates greatly improves a number of important device properties when compared with heteroepitaxial GaN on SiC, sapphire, or silicon. High-purity undoped GaN substrates, with a thickness of about 450 μm , were prepared by hydride vapour phase epitaxy at Kyma. A typical dislocation density in these substrates is $5 \times 10^6 \text{ cm}^{-2}$.

We investigated the N- and Ga-polar faces with the surface photovoltage (SPV) technique using a Kelvin probe attached to an optical cryostat. Experiments were conducted either in vacuum or pure oxygen. Additionally, photoluminescence (PL) spectra taken at different temperatures were analyzed for the same samples. Some of the surfaces were mechanically polished (MP), while others were epi-ready after a chemical-mechanical polish (CMP).

From the SPV measurements, the band bending in a sample having both surfaces treated with the CMP method was calculated to be about 0.74 and 0.57 eV for the Ga- and N-polar surfaces, respectively. SPV transients under continuous illumination with UV light indicated that the N-polar surface was more prone to the photo-induced adsorption of negatively charged oxygen species. The restoration of the SPV after ceasing the UV illumination showed that the SPV from CMP-treated surfaces behaved as predicted by a thermionic model, whereas the SPV from MP-treated surfaces restored with a much faster-than-predicted rate. This result could be interpreted by the hopping of charge carriers in the highly-defective near-surface layer of the MP-treated samples.

The PL from the CMP- and MP-treated surfaces had quantum efficiencies of about 1% and 10^{-5} %, respectively, indicating that for the MP surfaces the PL is quenched by defects located near the surface. Thus, both the PL and SPV data indicate that there is a higher density of near-surface defects for the MP-treated surfaces. In order to estimate how thick the defective region of the MP-treated surfaces is, the sample was etched with the inductively-coupled plasma to different depths. The quantum efficiency of PL increased by several orders of magnitude after a 0.7 μm -thick layer was etched off.

In the PL spectra from the defective region of the MP-treated surfaces, two defect-related bands dominate: the red and green bands peaking at 1.85 and 2.35 eV, respectively. These bands have been previously observed in undoped GaN layers grown in extremely Ga-rich conditions by the molecular beam epitaxy method. These bands, labeled in our earlier studies as the RL2 and GL2 bands, exhibited unusual properties. In particular, they were quenched with an activation energy of just 120 meV. A possible origin of these bands will be discussed.

Properties of MOCVD AlGaN/GaN heterostructures grown on polar and non-polar bulk GaN substrates

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Metal organic chemical vapor deposition (MOCVD) is a well-established method for the fabrication of various electronic devices, including laser diodes, UV detectors and field effect transistors. In late 90s, this technique was used to grow first GaN diodes and other GaN-based devices. Up to now, the majority of GaN-based structures are grown on foreign substrates, including sapphire, silicon carbide, silicon, zinc oxide, and many other due to the lack of cheap and large size GaN substrates. Recent progress in the growth of bulk GaN substrates by the ammonothermal method [1] opens new opportunities in the area of the fabrication of GaN-based devices as well as their full commercialization. Truly bulk GaN substrates grown by this technique deliver both the target size and excellent quality not only to the GaN substrates but also to epistructures grown on these substrates [2].

In this work we present structural, optical and electrical properties of GaN epilayers and AlGaN/GaN MOCVD heterostructures obtained on polar and non-polar 10x10 mm², 1 and 1.5 inch bulk ammonothermal GaN (A-GaN) substrates. Presented results are based not only on a single-point measurement but also on mapping across sample area. In addition to the fundamental transition, contactless electroreflectance (CER) features related to optical transitions between excited states were clearly observed for GaN/AlGaN QWs grown on *c*-plane GaN substrates whereas such features were not observed for GaN/AlGaN QWs grown on *a*- and *m*-plane GaN substrates. This experimental result clearly shows that the polarization-related electric field leads to a quantum confinement of some extra states in the polar QW system. Such states are not confined in the non-polar QW and, therefore, optical transitions between them are not detected, i.e., only the fundamental transition is observed in CER spectrum of the non-polar QW.

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Quantum-confined Stark Effect and Carriers Localization in $\text{Al}_{0.3}\text{Ga}_{0.7}\text{N}/\text{Al}_{0.4}\text{Ga}_{0.6}\text{N}$ Quantum Wells of Different Morphology

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AlGa_xN alloys are essential for semiconductor ultraviolet (UV) optoelectronics but enormous dislocation densities in these materials impede the achievement of a high emitting efficiency in the UV-emitters. The localization of carriers can improve situation and this effect has been demonstrated in the both bulk AlGa_xN layers and double heterostructure UV LEDs grown by plasma assisted molecular beam epitaxy (PA MBE) at the nitrogen rich conditions [1]. AlGa_xN quantum well (QW) heterostructures are also used in UV-emitters but their optical properties are drastically affected by spontaneous and piezoelectric polarization fields allowed by the symmetry of the AlGa_xN wurtzite films. Especially disadvantageous is the quantum-confined Stark effect (QCSE) caused by the intrinsic electric fields since it reduces oscillator strength and causes red shift of the involved interband optical transitions in the emitting QW. In this work we demonstrate that localization of carriers by enhanced potential fluctuations in the QW layer can substantially suppress the QCSE, thus increasing the emission quantum yield and decreasing the emission wavelength.

Investigated samples with an $\text{Al}_{0.3}\text{Ga}_{0.7}\text{N}/\text{Al}_{0.4}\text{Ga}_{0.6}\text{N}$ single QW (SQW) were grown by PA MBE on c-sapphire substrates and thick (1 – 1.5 μm) AlN buffer layers. The buffers of different samples were grown in either slightly metal-rich or strongly N-rich conditions to obtain two- or three-dimensional growth surface morphology, respectively. Various tricks, e. g. insertion of a short-period superlattice (SL) in buffer layers, were used to reduce density of threading dislocations. Following the AlN buffer, $\text{Al}_x\text{Ga}_{1-x}\text{N}$ layers of 400 and 500 nm width were grown with the sequentially diminishing aluminum content ($x = 0.77$ and $x = 0.4$). Within the latter layer a SQW was fabricated by a sub-monolayer digital-alloying technique at the distance of 75 nm from the surface. In this technique, the SQW is formed as a 6-nm-wide $\{\text{AlGa}_x\text{N}/\text{Ga}_x\text{N}\}_8$ SL with the average composition $\text{Al}_{0.3}\text{Ga}_{0.7}\text{N}$.

The structures were investigated by transmission electron microscopy (TEM) and photoluminescence (PL) spectroscopy. TEM images confirm basically plane morphology of the samples with the buffer grown in the metal-rich conditions and rough one induced by the N-rich growth conditions. PL spectra of the heterostructures with rough morphology demonstrate suppression of the QCSE, as compared to the samples grown in the planar mode, that is evidenced by more than an order of magnitude enhanced emission intensity and its blue shift up to 150 meV. The effect can be explained by emergence of an efficient fluctuating localization potential with the typical localization length comparable to the exciton Bohr radius, as a result of compositional modulations in the AlGa_xN SQW, enhanced by the three-dimensional growth mode. Such heterostructures with improved external quantum efficiency can be employed for manufacturing deep UV LEDs with increased emission efficiency.

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Optical saturation of intraband absorption of GaN/AlN quantum-dot waveguides at 1.55 μm

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The next generation of photonic devices should evolve to an all-optical approach to fully exploit the bandwidth of the optical fiber at 1.55 μm in optical communication networks. Particularly, all-optical switching devices require ultrafast response, low switching power, and high contrast ratio. Low-dimensional semiconductor structures, such as GaN/AlN-based quantum wells (QW) and quantum dots (QD), have demonstrated to be a suitable approach to attain the required specifications due to the short recovery time (~ 100 fs) of their intraband transition at 1.55 μm [1]. QD-based devices are a promising alternative to reduce the relatively large saturation intensity (in the range of a few tens of $\text{W}/\mu\text{m}^2$) present in the QWs [2]. In this work, we present the design, fabrication, and characterization of III-nitride QD waveguides optimized for nonlinear-optical switching at 1.55 μm .

The structure under study consists of 0.6- μm -thick GaN guiding layer deposited by plasma-assisted MBE on a 1.1- μm -thick AlN lower cladding on sapphire. The active region consists of 3 periods of self-assembled GaN QDs, synthesized by deposition of 0.75 nm of GaN under N-rich growth conditions followed by 15 s growth interruption, embedded in 3 nm AlN barriers. The electronic properties of the heterostructure were modeled using the Nextnano3 8-band k.p Schrödinger-Poisson solver. A GaN buffer layer of 25 nm was deposited below the QDs to avoid the polarization-induced depletion of the active region. High-resolution TEM images present QDs with a height of 1–1.5 nm and a base diameter ~ 7 nm, with the QDs not vertically correlated. Waveguide (WG) optical modes were calculated at $\lambda_0 = 1.55$ μm using the commercial finite-element based mode solver RSoft BeamProp. The single mode condition was determined as a function of ridge width, r_{width} , and height. An etching depth of 350 nm is enough to avoid slab guiding, and quasi-single mode propagation can be considered for WGs with $r_{\text{width}} = 5$ μm . Taking into account these design parameters, the estimated modal effective area of the device is ~ 4 μm^2 . Waveguides with $r_{\text{width}} \sim 5$ μm and 2 mm long were defined by optical lithography and ICP etching. Optical measurements were carried out to obtain the transmittance of the fabricated devices. A strong nonlinear saturation of the intraband absorption at 1.55 μm is demonstrated for low input powers for these materials. In particular, a contrast ratio of 12 dB for input pulse energy of less than 1 pJ is measured with 100 fs pulses.

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Continuous wave OSL in bulk AlN single crystals

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It is well known that single crystals and nanopowders of aluminum nitride efficiently store ionizing radiation energy of corpuscular and photon origin. At further selective or combined photo-thermal treatment optically or/and thermally stimulated luminescence are observed, i.e.: OSL and TL, respectively [1]. In this case recombination and radiation-optical properties of different structural states of aluminum nitride result from different intrinsic and impurity defects. In particular, high sensitivity to β - and UV-radiation and formation of dosimetric TL- and OSL-response of detecting matrixes based on AlN are caused by radiation-activated mechanisms occurring in oxygen complexes including vacancies in anion and cation sublattices: V_{N^-} , V_{Al} , O_{N^-} and $(V_{Al}-O_{N^-})$ -centers [2]. The aim of the present study is to analyze kinetic mechanisms of optically stimulated luminescence in bulk AlN single crystals with β -radiation dose being varied.

Aluminum nitride single crystals (Nitride Crystals Ltd.) obtained by sublimation–recondensation method have been studied. Oxygen content of the samples under study did not exceed 10^{18} cm^{-3} . The final samples were discs 15 mm in diameter and 0.25 – 0.50 mm thick with epi-ready surface. The AlN single crystals were exposed to β -radiation from the $^{90}\text{Sr}/^{90}\text{Y}$ source with $D = 10^{-3} - 1 \text{ Gy}$. OSL was measured with original spectrometric installation. Green DPSS laser (150 mW, 532 nm) was used to stimulate continuous wave OSL (CW-OSL). Sample emission was registered in the 3.25 eV band and $\text{FWHM} = 0.27 \text{ eV}$ by a photomultiplier FEU-39A with optical filters set.

As a result of the measurements, time dependences of optically stimulated glow in the irradiated single crystals were obtained, with the dose being varied. It is shown that after irradiation intensive afterglow of the samples is observed which overlaps the OSL signal under study. The experimental data are analyzed in terms of general-order kinetics formalism. It is shown that decrease of the OSL intensity is caused by two exponential processes with decay time $\tau \approx 32$ and 212 s . Photoionization cross section of the trap centers responsible for the registered OSL emission has been evaluated in the crystals under study: $\sigma \approx 5 \cdot 10^{-19}$ and $7 \cdot 10^{-20} \text{ cm}^2$.

Basic mechanisms of the OSL in the samples under study have been analyzed using independent literature data and investigation results for the associated TL processes. Possibilities of practical application of the AlN single crystals containing oxygen has been demonstrated for solid state dosimetry of beta irradiation in the CW-OSL regime and with accounting for induced afterglow.

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Effect of the nitridation on sapphire surface polaritons

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Due to the large mismatch of lattice parameters while epitaxial growth of III-nitrides layers on sapphire substrates (Al_2O_3) there are various negative effects. In order to reduce these effects, a heated sapphire substrate undergoes to nitride by soaking in a stream of ammonia. Under the action of ammonia thin (several monolayers) layer of AlN is formed on the surface of sapphire, which leads to higher quality of the created film. Nitridation process is controlled by reflection high-energy electron diffraction (RHEED). We have investigated the effect of annealing the sapphire substrate on subsequent nitridation process. To study so thin layers very sensitive techniques are necessary.

Surface polaritons (SP) are non-radiative interface electromagnetic modes. They propagate along the interface between 2 media, if one of these media is absorbing (metal or dielectric with the strong absorption bands). For thin films or multilayer structures SPs on different interfaces interact resulting in SP shift and splitting. We have compared external reflectivity spectra and SP spectra obtained using attenuated total reflection (ATR) technique for 3 samples:

- 1 – sapphire substrate,
- 2 – sapphire, annealed in vacuum at temperature 900°C,
- 3 – sapphire, first annealed, then nitridated in ammonia flux.

The measurements of the external reflectivity and ATR measurements in Otto configuration have been done using IFS66v (BRUKER) infrared Fourier-transform spectrometer. The reflectivity unit was used for the external reflectivity spectra at near normal incidence. The ATR unit with KRS-5 prism was used for various angles of incidence (20-60 degrees in the prism) in p-polarized light to study transverse magnetic SP. The angular dependences of the ATR minima give us the SP dispersion curves.

The samples external reflectivity spectra measured at near normal incidence exhibit almost no difference. On another hand ATR spectra allow to discriminate the samples. As far as the SP electromagnetic field concentrates near the interfaces, these excitations are more sensitive to the surface state or to the presence of thin films, then external reflectivity or transmittance techniques. The position of the minimum near 700 cm^{-1} depends clearly on the sample treatment and the change of the SP dispersion curve allows estimate the parameters of the transition layer obtained at the sapphire nitridation.

Characterization of MBE grown AlGa_xN layers heavily doped using silane

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Due to the wide and direct band gap Al_xGa_{1-x}N alloys have a wide application in light emitting diodes, high electron mobility transistors, high density optical storage devices, ultraviolet emitters and photodetectors and, especially, in solar-blind photodetectors fabrication. Precise conductivity control of AlGa_xN layers is needed to fabricate multiplayer device structures. Silicon is usually used in molecular beam epitaxy (MBE) for doping of AlGa_xN layers. However, in ammonia MBE a silicon nitride film covers silicon changing the silicon flux in uncontrolled fashion. In present work silane was introduced as silicon dopant during growth of AlGa_xN layers by ammonia MBE.

The Al_xGa_{1-x}N layers with wide range of AlN molar fraction ($0 < x < 0.6$) were grown by ammonia MBE on (0001)-oriented sapphire substrates. The thickness of the layers was about 1.1-1.3 μm. In order to characterize the structural, electrical and optical properties, atomic force microscopy, X-ray diffraction, Hall, Raman, photoluminescence (PL) and cathodoluminescence (CL) measurements were performed.

With increase of SiH₄ flux an electron concentration in GaN layers linearly rises up to $n=1 \times 10^{20} \text{ cm}^{-3}$, while a electron mobility gradually decreases down to $\mu=120 \text{ cm}^2/\text{Vsec}$. The very close electron concentrations were obtained from the shift of phonon-plasmon coupled modes in Raman spectra of GaN layers. The electron concentration and electron mobility of about $n=4 \times 10^{19} \text{ cm}^{-3}$ and $\mu=60 \text{ cm}^2/\text{Vsec}$, as well as $n=2 \times 10^{19} \text{ cm}^{-3}$ and $\mu=15 \text{ cm}^2/\text{Vsec}$ were reached in Al_{0.3}Ga_{0.7}N and Al_{0.6}Ga_{0.4}N layers, correspondingly. It should be noted that in the Al_{0.3}Ga_{0.7}N layers the electron concentration saturates, whereas in Al_{0.6}Ga_{0.4}N layers the electron concentration decreases after running up to the maximum values. Phonon-plasmon coupled modes have been observed in a few Al_{0.3}Ga_{0.7}N layers. The PL intensity of GaN layers linearly enhances with increase of the SiH₄ flux. In happens in spite of the fact that the full-width at half-maximum of the GaN(0002) and (10-15) double-crystal X-ray rocking curves, which proportional to the threading dislocations density, increases with increase of the SiH₄ flux. In room-temperature CL spectra of Al_{0.6}Ga_{0.4}N layers two bands were observed. The CL intensity of edge band quickly saturates, whereas the intensity of "green" band grows with increase of the SiH₄ flux.

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Characterization of nitrides based LED materials using dynamic SIMS

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Improving the manufacturing yield in LED highly competitive market requires process control strategy similar to what has been applied to IC manufacturing for years. Nitrides based LED technology is based on multilayer structures grown by means of EPI tools (MOCVD), doped with species like Mg, Si, Zn, and with electrical properties suffering from contamination like H, C, O.

Like for Si technology development for ICs, dynamic SIMS is a key tool for R&D of future LED devices, as it provides depth profiles with excellent detection sensitivity for dopants and impurities, while keeping high analysis throughput. SIMS is particularly attractive for the analysis of light elements, for which the detection limits surpass the capabilities of competing techniques

Therefore, dynamic SIMS technique can be extremely useful to solve various problems:

- Doping concentration and distribution (Be, Si, Mg, Zn)
- Impurity control (H, C, O, metals,...)
- Layer structure control
- Layer interface quality (junction impurities, diffusion of contaminants,...)
- Film composition

thus reducing the R&D cycle time and enhancing the production yield.

Data obtained using CAMECA's LED dedicated magnetic sector SIMS will be presented. This instrument is based on a double focusing mass spectrometer, and provides very high performance in terms of sensitivity, depth resolution and mass resolution which is mandatory for these applications. Depth profiling and detection limits obtained for light elements as well as for Mg and Si dopants will be shown.

Self-organized defect control during GaN homo-epitaxial growth on nano-structured substrates

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We report on implementation of control over defect formation in GaN films grown by Hydride Vapor-Phase Epitaxy (HVPE) on free-standing GaN substrates with nano-structured bulk of the material. For the first time, a mechanism is described which is responsible for collective interaction of structural defects, and allows for excluding substrate dislocations from the sources of threading defects in homo-epitaxially grown GaN films.

The nano-structured substrates were fabricated using anodization, which allows for forming porous structure in the bulk of GaN wafer. Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) studies showed that high-temperature treatment during epitaxy lead to peculiar modification of nano-porous structure in the substrate. Namely, formation of spheroid-like macro-pores along substrate dislocation lines was observed. This resulted in segmentation of dislocations. As it turned out, all of the dislocation segments were pinned at the inner surfaces of the pores, and could not propagate into the growing GaN films. Thereby, the density of threading dislocations in epitaxial films was much lower than that in the initial GaN wafers. In particular, TEM analysis of initial free-standing GaN wafers showed that they had density of dislocations of the order of $5 \times 10^6 \text{ cm}^{-2}$. At the same time, dislocations were not found in GaN films grown on substrates with porous structure. Taking into account the resolution limit of the method, one could estimate dislocation density in the films as 10^5 cm^{-2} .

We believe that the results obtained in this work can assist in developing new solutions of tasks related to fundamental and applied materials science, which address new methods of control over the structure and properties of semiconductor materials. On the other hand, these results have real practical value, as reduction in dislocation density in GaN films down to 10^5 cm^{-2} provides a defect density level required for further development of GaN-based lasers.

Low Energy Electron Beam Induced Ga-Vacancy Activation in Gallium Nitride Films

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Gallium nitride materials are thought to be extremely resistant to different types of radiation [1]. However, reduced cathodoluminescence and enhanced dislocation mobility have been observed even with low energy electron beam (e-beam) irradiation [2]. Low energy e-beams are frequently used in characterization and processing of GaN based samples. Therefore, more insight on the properties of e-beam irradiated GaN is needed to determine the effect of low energy irradiation.

In this work MOVPE grown GaN films were exposed to 5-20 keV e-beam using exposure doses of 0 – 500 $\mu\text{C}/\text{cm}^2$. The irradiation was done by sweeping a focussed e-beam (spot diameter 2 nm) on the sample surface, with maximum momentary current density reaching up to $10^5 \text{ A}/\text{cm}^2$. The films were analysed with photoluminescence measurements (excitation by 325 nm He-Cd laser) and positron annihilation spectroscopy. As we have reported before, exponential reduction of the photoluminescence intensity on increasing irradiation dose was observed [3]. The optical degradation increased with decreasing e-beam energy. This was associated with tighter confinement of the low energy e-beam energy dissipation profile near the surface, within the laser absorption depth. With increasing e-beam energy, the energy dissipation profile broadens and is shifted further from the surface.

Positron annihilation spectroscopy was used to study the cause of optical degradation. Measurements revealed a significant increase in the observed density of in-grown Ga-vacancies (V_{Ga} density up to $2 \times 10^{17} \text{ cm}^{-3}$). The vacancy density correlated well with calculated e-beam energy dissipation profiles and the level of optical degradation.

After irradiation of 5 keV e-beam with a dose of $100 \text{ C}/\text{cm}^2$ the luminescence intensity of the GaN samples was reduced to approximately 25% of the original intensity. The recovery of the optical degradation of the irradiated GaN films was achieved by thermal annealing at 550°C . Annealing for 10 min in both H_2 and N_2 ambient recovered the PL intensity in average to 50% of the original. For another set of GaN samples, with identical irradiation parameters, anneal of 30 min further improved the intensity from 20% to 55% of the original intensity under both gases.

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Doping effect on optical properties of freestanding bulk GaN

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Further development of GaN devices for efficient high power applications demands epitaxial growth on native GaN substrates with low defect and impurity densities. Control of the dopant concentrations in bulk GaN is important for the deposition process since it affects infrared absorption. Also, high impurity concentrations in the substrate may result in undesirably enhanced doping levels in the active layer of devices.

We have studied how intentional doping by silicon, oxygen and carbon affects low-temperature luminescence properties of freestanding thick GaN wafers. Samples were grown by halide vapor phase epitaxy (HVPE) at 1000 °C on 2 inc. bare sapphire wafers or with a 1 μm thick GaN seed layer grown by metal-organic vapor phase epitaxy. HVPE GaN layers with thickness of 1-2 mm were usually partly or completely self-separated from the sapphire. O₂ and C₂H₂ diluted in nitrogen gas were used for doping by oxygen and carbon, respectively. Mixture of silane (SiH₄) and hydrogen was a source for silicon doping. Average impurity concentrations were estimated from secondary ion mass spectrometry and the degree of activation was determined from electrical measurements.

We have found that the concentration of silicon in GaN can be reduced by one order of magnitude using a moderate intentional doping of oxygen. A strong band gap narrowing of ~6 meV was observed with increasing doping in the studied samples. The low temperature photoluminescence (PL) recombination time for donor bound excitons (DBE) was found to decrease with increasing donor concentration. A model assuming generation of DBEs by capturing of free excitons by neutral donors explains the experimental results at low temperature. From fitting the experimental lifetime for the DBE a lower limit for the radiative time and the upper limits of the effective capture cross-section of bound excitons were estimated.

In-plane view cathodoluminescence images revealed that the lateral doping distribution in bulk GaN can be non-uniform, especially if the growth was done on bare sapphire. In the later case hexagonal domains of different orientation with threading dislocations in the center violate the crystalline structure. Areas with high doping concentrations correspond to $\{11\bar{2}2\}$ facet of the domains, while areas with (0001) orientation, i.e. without domain inclusions, have a low concentration of dopants. This non-uniform doping distribution can be understood in terms of different incorporation probability of the impurities in different crystal planes during growth.

Role of extended defects in the transformation of InGaN/GaN multiple quantum well structure optical properties under low energy electron beam irradiation

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It was previously shown that the low energy electron beam irradiation (LEEBI) in the scanning electron microscope essentially influences the electrical and optical properties of InGaN/GaN multiple quantum well light emitting structures. It was observed that the InGaN related luminescence intensity remarkably increases during the first stage of LEEBI. The effect is more pronounced at beam energies, at which the excess carrier concentration in the quantum wells is higher. In all structures studied one or a few new bands blue shifted regarding the initial emission band were observed after irradiation. Low temperature CL measurements confirm that the initial emission band still exists together with new one. Investigations of LEEBI effect on the electron beam induced current (EBIC) signal dependence on beam energy allowed to conclude that LEEBI leads to a decrease in an effective recombination rate in the upper p-GaN layer, to a decrease of depletion region width (increase of donor concentration in the active layer) and to a small increase of excess carrier portion captured into quantum wells.

Investigations of inverted structures with a thick (about 3 μm) top GaN layer allow to show that the LEEBI effect is determined not by a surface reactions stimulated by LEEBI but by the bulk processes stimulated by the excess carriers created by electron beam. An increase of top GaN layer thickness leads to an increase of beam energy, at which LEEBI effect can be observed. Thus, the effective new emission band formation occurs at beam energies higher than 30 keV only. It is very important that while in the initial structure the InGaN related emission band is observed by the cathodoluminescence at beam energies higher than 25-30 keV, after LEEBI the new emission band can be observed even at 10 keV. The similar behaviour is observed on degraded structures. An analysis of results of EBIC measurements shows that even in initial structures a part of excess carriers reaches the depletion region through the thick GaN layer at low beam energy. It could be assumed that such carriers move along threading defects because it is well known that excess carriers can transport along charged extended defects on a long distances. Thus to explain the results obtained it could be assumed that new emission bands are formed in near defect regions and the excess carriers can reach these regions even when they are created far from the active layers. This means that the changes of quantum well optical and electrical properties stimulated by LEEBI occur preferentially near threading defects. Such conclusion can be confirmed by the more effective changes of quantum well electrical properties due to LEEBI near bright contrast defects revealed by the EBIC measurements.

HVPE growth of GaN in the semipolar direction on planar Si(210)

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At that work a layer of semipolar gallium nitride was grown on Si(210) substrate by hydride vapor phase epitaxy (HVPE). As a buffer a nanocrystalline layer of 3C-SiC was used. During this growth the nanocrystalline SiC nuclei form at a very low supersaturation referred to ‘quasiequilibrium’ growth of SiC nuclei as the solid state reaction occurs inside Si substrate rather than outside. A thin layer of hexagonal nitride aluminum (~100 nm) was grown by HVPE on a silicon carbide layer and then a layer of thick (~ 10 mkm) hexagonal gallium nitride, both in hydrogen atmosphere.

It has been found that anisotropic strain of the growing nitride layer changed the crystallographic orientation of its surface: with increasing layer thickness orientation of the surface changed sequentially as follows: (0001), (10-15), (10-14), (10-13) and, finally, (10-12). Rocking curve halfwidth of (10-12) antisymmetric reflections from the GaN layer was $w_q=25$ arcmin. In other words, in epitaxy of a layer under anisotropic strain blocks of the growing hexagonal crystal deviate from C-axis by as much as ~48 deg. GaN (10-12). Electron microscopy of the layer surface showed that its structure was characteristic of a layer grown in semipolar direction.

In photoluminescence spectra at 77 K of GaN layers well defined luminescence bands can be observed with maximums at $h\nu_{\max} = 3.46$ eV, 3.27 eV, 3.19 eV and 3.1 eV. A small peak at an energy of $h\nu_{\max} = 3.1$ is possibly due to cubic modification of c-GaN, because cubic gallium nitride can be present on 3C-SiC(100) planes of the layer in the initial growth phase.

Thus, a new method has been proposed and proved of growing epitaxial gallium nitride layers on planar Si(210) substrate with a 3C-SiC buffer layer along semipolar direction under conditions of anisotropic strain in the hexagonal nitride layer. It is established that the proposed approach to growing gallium nitride in semipolar direction on silicon substrates can be very useful for obtaining ‘templates’ for manufacture of structures used in nitride optoelectronics.

CHVPE GaN growth on SiC and Si

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Increasing demands for higher voltage, higher efficiency semiconductors are driven by the markets for alternative, clean energy such as solar and wind and by increasing trends toward all-electric and electric-hybrid vehicles. The wide bandgap semiconductors (SiC, III-nitrides) can exceed the performance of Si. In comparison to SiC, GaN has higher mobility at higher doping resulting in lower on-resistivity and higher efficiency. Since GaN is a direct bandgap semiconductor it has faster recombination than SiC and therefore lower switching losses. In contrast to SiC, the III-nitride family can form heterostructures resulting in a wider range of useful devices. However, GaN has lower thermal conductivity than SiC.

This can be mitigated by combining the two materials. It has been demonstrated by a number of researchers that low-defect GaN can be successfully grown on SiC. The most obvious example of this is Cree's high-performance blue LEDs on SiC substrates. Similarly, high performance metal-semiconductor (MS) junction (Schottky) devices with very high switching speed and nearly negligible operating and switching losses have been demonstrated with GaN. Higher power density operation can be achieved by GaN-on-SiC Schottky devices by utilizing the high thermal conductivity of SiC for efficient heat extraction.

A number of researchers have demonstrated that GaN can also be grown epitaxially on Si. The most widely known examples of this are Schottky diodes by Nitronex, IR and others and blue LEDs by Bridgelux, Latticepower and others. Higher quality GaN material can be obtained by using (111) Si substrates, although (001) Si substrates have been used also. This allows the high voltage, high switching speed and inherently low switching losses of GaN to be integrated with Si structures.

The epitaxial growth technique that has been used in the industry is MOCVD. Nitride Crystals specializes in CHVPE growth. This is inherently a much less expensive and higher growth rate technique than MOCVD. The raw materials have 10x lower cost and the growth rate is can be more 100x higher. Nevertheless, CHVPE has also been used to grow quantum wells with TDI as the best-known example of this. Additionally, TDI, NC and others have demonstrated that CHVPE can be used to grow the full range of material composition from AlN to InN.

We will present results of NC's development of AlGaIn and GaN growth on SiC and Si substrate.

Growth of GaN Nanorods on Si (111) substrate by Reactive Magnetron Sputter Epitaxy using a Liquid Ga as a Sputtering Target

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GaN is one of today's most important semiconductors used for applications such as blue and ultraviolet light emitting diodes and laser diodes, thanks to its large direct band gap of 3.4 eV at room temperature. One-dimensional nanostructured GaN materials, such as nanorods, nanowires and nanotubes, have attracted extensive interest due to their great prospects in novel nanotechnology applications as well as in fundamental physics.

Recently, various low dimensional GaN structures such as nanowire, nanorod, nanobelt, and nanosheet have been successfully synthesized by various deposition techniques such as simple gas reaction, arc discharge, laser ablation, chemical vapor deposition (CVD) and molecular beam epitaxy (MBE). Growth of GaN based nanostructures by direct current magnetron sputter epitaxy (DC-MSE) have never been reported before.

In this work we report, the growth of GaN (0001) nanorods on to Si (111) by reactive DC-MSE by sputtering a liquid Ga target in a mixture of N₂ and Ar. Different total pressures ranging from 4.5 mTorr to 10 mTorr were used with a nitrogen partial pressure varying from 2 mTorr to 10 mTorr. The growth temperatures from 800 °C to 1000 °C were investigated. The surface morphology, length and the diameters of the rods was studied by scanning electron microscope. It is observed that by changing the temperature and the N₂ partial pressure, the diameter of nanorods varies in the range of 80 nm to 35 nm and the length of the nanorods also varies from 500 nm to 1000 nm. Cross-sectional high resolution transmission electron microscopy has shown that the GaN nanorods have good crystalline structure. All the GaN nanorods are 0001 oriented along the growth direction but they have random in-plane orientation. The nanorods exhibit low temperature (4K) photoluminescence peak at 3.474 eV with a FWHM of 36 meV which is attributed to intrinsic GaN band edge emission. As DC-MSE is easily scalable, it enables large-scale growth of high quality GaN nanorods onto very large non-lattice matched substrates at ultra high vacuum conditions.

Improvement in crystalline quality of GaN prepared by pulsed sputtering by the use of SiN_x islands

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Recently, fabrication of nitride devices by low cost processes such as sputtering has attracted much attention. In fact, various nitride devices such as LEDs, solar cells, and HEMTs have been already demonstrated with the use of pulsed sputtering epitaxial growth process [1]. To take full advantage of high throughput sputtering process, the use of low cost large area substrates such as Si is highly desirable. However, GaN growth on Si substrates still suffers from formation of a high density of crystalline defects such as threading dislocations. In this study, we will report on a technique that allows us to improve crystalline quality of sputtering GaN epitaxial films on Si by the insertion of SiN_x islands.

Epitaxial growth was performed on Si(110) substrates with a pulsed sputtering deposition (PSD) apparatus. We chose the (110) plane of Si because it has a couple of advantages such as small lattice mismatches with group III nitrides along Si[100] direction and compatibility with the CMOS technology [2,3]. On top of 60-nm-thick AlN buffer layers, 800-nm-thick GaN films were grown with and without insertion of SiN_x. The GaN epitaxial films were characterized by the use of various techniques such as PL, RHEED, XRD, and AFM.

XRD measurements have revealed that the epitaxial relationship between GaN and Si(110) for all the samples is $[1\bar{1},100]\text{GaN} // [001]\text{Si}$ and $[0001]\text{GaN} // [110]\text{Si}$, which makes the lattice mismatch in this system minimum. We also found that the GaN films on the AlN/Si(110) structure show Ga polarity. We then inserted SiN_x after the growth of 150-nm-thick GaN. RHEED and AFM observations have revealed that SiN_x deposited on the surface of GaN forms a high density of amorphous islands with a diameter of approximately 20 nm and a height of 2 nm. We resumed growing GaN on top of SiN_x and found that the top GaN films show a clear streaky RHEED pattern for c-plane GaN, which indicates that the epitaxial growth of GaN proceeds. RHEED and SEM observations of the GaN surfaces before and after KOH etching led us to conclude that the polarity of the GaN films remains unchanged as Ga-polarity even after the insertion of the SiN_x islands. The FWHM values of x-ray rocking curves for 0002 and 10 $\bar{1}$,12 diffractions of GaN with SiN_x were 17 and 22 arcmin, respectively, while those without SiN_x are 20 and 28 arcmin, respectively. These results indicate that the insertion of amorphous SiN_x islands leads to improvement in the crystalline quality of PSD GaN films on Si(110) substrates probably due to the blocking of the propagation of threading dislocations from the nitride/Si heterointerface.

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Effect of silicon-nitride intermediate layer on self-aligned GaN nanorod growth on Si(111) surface

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Formation of spontaneously aligned crystalline nanorods on Si surfaces has been a challenge to achieve. There are numerous approaches reported in literature to grow GaN nanostructures such as use of AlN buffer layer, ELOG, VLS mechanism etc. Out of them surface modifications to make surface lattice compatible to GaN have attracted great attention, because of its process compatibility to MBE and ability to form the requisite template for initial nucleation of strain relaxed crystalline GaN nanostructure growth. We present here an approach to catalyst-free growth of GaN nanorods on Si(111) surface and show that their alignment depends on the nature of an ultrathin silicon nitride intermediate layer. While nanorods grown on bare Si(111) and non-stoichiometric silicon nitride interface are disoriented, those grown on single crystalline Si₃N₄ intermediate layer form a high density of c-oriented hexagonal shaped nanorods that form an ordered 'ab-ab-ab' hexagonal superstructure. The nanorod assembly shows high-quality crystallinity and optical emission properties. The self organization of the nanorods suggest that they may be nucleated at screw dislocations at the interface and grow spirally in the c-direction.

Growth of the thick GaN layers on Si substrates

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Silicon is a desirable substrate for growing GaN because of its high crystalline quality, large size, and low cost. However the large mismatches in the lattice parameters (16.9%) and thermal-expansion coefficients make difficult to growth thick GaN layer on Si substrates.

In this work we have investigated the deposition of GaN on Si (111) substrates. Growth was carried out using low-pressure MOCVD reactor (AIX2400G3HT) under hydrogen ambient. Trimethylaluminum (TMAI), trimethylgallium (TMGa) and ammonia (NH₃) were used as precursors. The pressure in the reactor was maintained at 100 mbar for AlN and 200 mbar for GaN. The temperature was differed in range 1000-1130 °C. The V/III ratio was changed in a range from 100 to 1000.

The structural quality of the epilayers was characterized by X-ray rocking curve measured in reflection (0002) by means of double-crystal diffractometer «Vektor». The morphology of the epilayers was evaluated by interferometer of incoherent light «NV6200». The PL spectrums were measured by «RPMSigma» at room temperature. «In situ» analysis was carried out by means of «LayTec EpiCurveTT».

Low temperature GaN nucleation layer (NL) is not suitable for growing GaN on Si because of Si surface is deteriorated by GaN NL during heating to the GaN growth temperature. In this work we used AlN seed layer with subsequent growth of Al_{0.6}Ga_{0.4}N and Al_{0.2}Ga_{0.8}N intermediate layers. After that we grew undoped GaN with thickness of 800 nm. The surface of GaN layer looks like a mirror with many cracks observed by optical microscope. The reason of these cracks consists in large mismatch in thermal-expansion coefficients ($5.59 \cdot 10^{-6} \text{ K}^{-1}$ for GaN and $3.59 \cdot 10^{-6} \text{ K}^{-1}$ for Si). Strong concave bowing of the wafer is observed during cooling from growth temperature to room temperature. The concave curvature is changed from 60 to 190 km⁻¹ during cooling.

In the next sample we used the same AlN/AlGaN buffer layer. To exclude cracks and increase the thickness of GaN we insert a thin low temperature AlN interlayers after every 800 nm of GaN growth. The single AlN intermediate layer can introduce sufficient compressive stress to the top GaN layer and thus counterbalances the thermal tensile stress in the nitride layers imposed by the silicon substrates. Optimizing the temperature and thickness of AlN layers we can grow GaN with overall thickness about 2.5 μm. The surface of thick GaN sample looks like mirror and we don't observe cracks on the wafer. Results of samples measurement is showed in the table.

Table 1. Results of samples measurements.

Sample number	Thickness of GaN, μm	X-ray (0002) FWHM, arcsec	PL, RT, λ, nm	PL, RT, FWHM, nm	RMS, nm
N1	0.8	602	365.6	9.9	4
N2	2.5	515	366	15.7	8

Activity modulation MEE growth of 2H-AlN on Si(111) using double buffer layer grown by PA-MBE

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A radio frequency inductively coupled plasma (rf-ICP) discharge of nitrogen gas is a most suitable way to produce nitrogen atoms to grow group III nitride semiconductors using plasma-assisted molecular beam epitaxy (PA-MBE). The rf discharge has two modes of discharge, one is high brightness (HB) discharge mode, which produces chemically active dissociated nitrogen atoms (N+N*) and physically active excited nitrogen molecules N₂*, where N is the ground state nitrogen atoms and N* is the excited state nitrogen atoms, and the other is low brightness (LB) one, which produces only physically active N₂*. These two modes are able to control by rf power. Periodically controlled exposure of Al, (N+N*) and N₂* fluxes make possible an activity modulation migration enhanced epitaxial (AM-MEE) growth. An interface reaction epitaxy (IRE) of beta-Si₃N₄ on Si(111), for which uniform indirect exposure of the active (N+N*) atoms is used, is the most effective method for a large size Si wafer. Successive Al exposure to the surface of the beta-Si₃N₄ produces IRE-AlN. Both layers, IRE-AlN/beta-Si₃N₄/Si could operate as a double buffer layer (DBL) [1-4] for AM-MEE growth of group III nitride semiconductors.

Hetero-epitaxial 2H-AlN thin films of 60 nm to 200 nm thickness on Si(111) were have been grown by the AM-MEE PA-MBE method. The influence of a DBL, which was formed in a MBE chamber, before the AM-MEE growth of 2H-AlN thin films on Si(111), was studied to improve the crystallinity of AlN films. For the growth of the DBL, the optimization of the cleaning method of Si surface and the growth condition of beta-Si₃N₄ was studied. The interface roughness of DBL as a layer of two layers of AlN and Si, AlN/DBL/Si was characterized by grazing incidence X-ray reflectivity (GIXR) to evaluate the relationship between the interface roughness and grown AlN films. The surfaces were observed by AFM.

Changing nitridation temperature of Si(111) in a MBE chamber, the thickness of beta-Si₃N₄ increased from 0.18 nm to 0.35 nm at from 830°C to 680°C, because of due to the change relation of between the surface density of (N+N*) atoms due to and the substrate temperature. By increasing the thickness of the AlN from 60 nm to 200 nm The crystallinity was improved from 53.9 arcmin to 51.7 arcmin, obtained by the measurement of rocking curve of omega full width at half maximum (FWHM) for AlN (0002) peak of X-ray diffraction, by increasing the thickness of the AlN from 60 nm to 200 nm. On the other hand, the surface roughness of AlN did not depend on the thickness of the AlN films obtained by fitting results of GIXR curves.

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MOVPE growth of GaN wires on Si(111) for LED applications

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The fabrication of planar LEDs on Si substrates is challenging due to the large lattice and thermal dilatation coefficient mismatches between GaN and Si. In MOVPE, The growth of thick and complicated AlGaIn-based buffer layer is required to limit the formation of extended defects in the GaN layer.

In this work, we demonstrate the fabrication of operating core/shell LED on GaN wires grown on Si(111). A thin AlN buffer layer allows a backside electrical contact that considerably simplifies the device processing.

The MOVPE growth of the n-doped GaN wires on Si(111) occurs in a similar manner to that on sapphire [1], leading to the formation of c-oriented wires. The epitaxial relationship of the wires with the Si substrate will be shown by XRD and cross-section TEM images to enlight the role of the AlN layer.

The active part of the LED, consisting of five InGaIn/GaN multi-quantum wells (MQWs) is grown on the standing wires in a core/shell geometry. Their photoluminescences reveal a main contribution between 400 and 450 nm controlled by the thickness and composition of the InGaIn wells.

For the complete LED structure, a p-GaN shell is grown around the MQWs. Using a backside contact and a conductive tip, electroluminescence measurements on single structures reveal a strong luminescence around 420 nm at room temperature.

The Contact of an assembly of wire-based LEDs on Si [2] is consistent with these results and exhibits the same strong electroluminescence at 420 nm.

Furthermore, some recent improvements of electrical properties of such LEDs on Si substrate will be presented.

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Self-induced MBE growth of GaN nanowires on Si substrates with thin Al₂O₃ buffer layer

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We report on self-induced growth of GaN nanowires (NWs) by plasma assisted MBE (PAMBE) on (111) silicon substrates with a thin Al₂O₃ buffer. Riber Compact 21 system equipped with elemental source of Ga and an Addon RF plasma source of active nitrogen was used for the growth. Before the growth freshly etched Si substrates were covered by a thin amorphous Al₂O₃ buffer layer deposited by atomic layer deposition at temperature of 85°C. Thickness of buffers used varied in the 2 nm – 15 nm range. Then, growth of nanowires started at ~750 °C under highly nitrogen-rich conditions. No catalyst was used to induce nucleation of NWs. Also the reference sample of GaN NWs was grown under similar conditions on bare Si(111) substrate that was exposed to a nitrogen flux for nitridation before epitaxial growth.

SEM studies of the samples showed that all growths resulted in a dense ensemble of ~370 nm long NWs with average diameter of ~40 nm. The wires were homogeneously distributed and well oriented with the c-axis being perpendicular to the substrate. XRD technique was used to analyze orientation of GaN NWs lattice relative to that of the substrate. By measuring X-ray pole figures of the GaN 00.4 and 01.2 reflections we observed high coherency of twist and tilt of NWs in the sample grown on nitridized Si(111) without the Al₂O₃ buffer. Moreover, an *epitaxial relationship* between GaN and Si(111) lattices, the same as commonly observed in planar GaN on Si(111), i.e. (0001)GaN || (111)Si, <-1-120>GaN || <1-10>Si was found despite presence of ~2 nm thick film of amorphous Si_xN between GaN and the substrate, as detected by TEM. In the samples grown on substrates with the Al₂O₃ buffer this epitaxial relationship was lost, even in the sample with the thinnest (2 nm) buffer used. Still a nearly isotropic angular distribution of the tilt of the NWs with the average tilt value smaller than ~2.5 deg was observed. However, the X-ray pole figure of the GaN 01.2 reflection revealed a random twist orientation of GaN NWs grown on the Al₂O₃ buffer layer. This finding clearly indicates that the preparation recipe, and thus the structure of a thin layer on which GaN NWs nucleate, strongly influences their ordering on the substrate.

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InGaN-based heterostructures for solar cells

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Nitride semiconductors are nowadays widely used in optoelectronic devices such as light emitting diodes, lasers and photodetectors. $\text{In}_x\text{Ga}_{1-x}\text{N}$ has been shown to be a good candidate for solar energy conversion since the material forms a solid solution with a band gap that can be tuned from 3.4 to 0.7 eV by altering the In-composition [1]. This range corresponds to the solar spectrum making $\text{In}_x\text{Ga}_{1-x}\text{N}$ alloys a promising candidate for radiation-resistant multi-junction solar cells.

Several challenges need to be overcome in producing high-quality InGaN materials. First, the large lattice mismatch of 11% between InN and GaN leads to a miscibility gap, which can cause phase separation in the multiple quantum wells (MQWs). Second, the low temperature growth of InGaN induces impurity incorporation and morphological defects, generating nonradiative recombination centres. Third, a strong built-in piezoelectric field induced by the significant strain for high indium content InGaN on GaN leads to a local separation of electrons and holes toward the opposite side in QWs. Nonetheless high quantum efficiency can be obtained using InGaN QWs [2].

In this work, we report on the characterization of series of samples grown on c-plane sapphire substrate of changing the number of MQWs (5, 15, 30) and the In-content (till 20%). The solar cells structures consisted of an unintentionally doped GaN template layer, followed by a Si-doped n-GaN layer before the MQWs. P-GaN layer on the top was made intentionally rough in order to decrease the reflection at the top p-GaN layer and increase the total light absorbed by the solar cell [2]. The morphology and physical features (structural, optical, electrical) of the samples were investigated by scanning electron microscopy (SEM), X-ray diffraction (XRD) including θ -2 θ scan, atomic force microscopy (AFM) and photoluminescence spectroscopy (PL).

The next step of this work is focused on the growth of solar tandem cells structure on the Si substrate (111). We proposed to grow In-rich InGaN layer on silicon substrate by Molecular Beam Epitaxy and the MQWs and p-GaN layer by Metal-Organic Chemical Vapour Deposition to take the advantages of these two methods. In-rich MQWs will be reached by optimizing the different parameters: the substrate temperature, Tri-Methyl flow, V/III ratio and the growth rate. The first results will be shown and discussed.

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Growth and deep level defect evaluation of InGaN films for the application of photovoltaic devices

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We have grown thicker InGaN films to apply them to an active layer in photovoltaic device. We have demonstrated the enhancement of conversion efficiency in the solar cell by inserting ultra-thin AlN layer between p- and i-InGaN layers [1]. In order to further improvement, the deep level defects in the films and at the interface junctions must be evaluated.

InGaN films and p-i-n structure were grown on the various templates by metalorganic chemical vapor deposition (MOCVD). Schottky or p-i-n junctions were fabricated, and I-V characteristics were measured in dark and under the illumination. Deep level defects were evaluated by using the various photo-capacitance techniques [2]. The profile of junction in depth were attempted to be analyzed by changing bias voltage during the measurements.

In this study, we are going to show the points to improve the photovoltaic properties with respect to the correlation of deep level defects.

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Fabrication of Zn-doped GaN-Pillars as Potential Sources for Single-Photon Emission

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Since quantum communication based on the polarization of photons has been demonstrated the interest in single-photon sources increases continuously. A very common single-photon emitter with appropriate count rates and excellent stability at room temperature is the nitrogen-vacancy (NV) center in diamond. However, due to doping problems this center can only be excited optically, but for the implementation of a widespread communication network based on the polarization of photons, electrically driven single-photon sources are required. This might be realized by a LED with two single quantum states within the active region. These quantum states can be provided either by quantum dots, e.g. InAs embedded in GaAs, or by single ions, when the mean distance between two ions is relatively large so that they can be excited separately.

At present GaN is the material of choice for commercial LEDs in the blue- and UV-spectral range. Furthermore, Zn in GaN creates a deep acceptor level about 0.3 eV above the valence band and shows a broad emission in the blue spectral range around 2.9 eV when co-doped with Si. This emission is attributed to donor-acceptor transitions at low temperatures and to electron-acceptor transitions at elevated temperatures. The internal quantum efficiency of the blue emission exceeds 90 %, making Zn in GaN a potential candidate for single photon emission [1].

In this contribution, we will focus on the fabrication of AlGaN / GaN:Zn,Si / AlGaN heterostructures with low Zn concentration using metalorganic chemical vapor deposition (MOCVD). Furthermore, the samples were patterned either by photolithography and dry etching or by ion-beam milling to fabricate 3-dimensional pillars with a reduced number of Zn ions in each pillar. We will discuss the influence of doping level, diameter and pitches of the pillars on the emission spectra with regard to the applicability of Zn-doped GaN for single-photon emitter.

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Structural characterization of epitaxial GaN wires: polarity determination and dislocation bending

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In the last few years, nanowires have attracted considerable attention for the growth of defect- and strain-free heterostructures and for their integration onto almost any substrate, including amorphous and even plastic materials. These two properties are especially interesting in the case of GaN since it is well-known that the performances of III-nitrides devices are limited by their high density of structural defects, in particular dislocations.

In this work, we will present a transmission electron microscopy (TEM) study of GaN microwires (typical diameters range from 500nm up to 5 μ m) grown by Metal-Organic Vapor Phase Epitaxy (MOVPE) on sapphire substrates [1]. First, we will discuss the polarity of the GaN wires, which was determined by convergent beam electron diffraction (CBED) within a TEM and confirmed by wet chemical etching. These complementary measurements indicate that MOVPE-grown GaN wires on sapphire show systematically the coexistence of Ga- and N-polar domains. Further TEM observations indicate that the inversion domain boundaries, which separate the regions of different polarities, nucleate at the sapphire/wire interface. The presence of the two polarities is expected, given that sapphire is not a polar substrate, and is consistent with previously published findings on GaN thin films grown by MOVPE on sapphire [2].

Furthermore, focused ion beam preparation enabled us to study complete GaN microwires, more than 20 μ m long, from the wire/sapphire interface up to the GaN wire top. We will show that, although dislocations are generated at the wire/substrate interface, they all bend after several hundreds of nanometers resulting in dislocation-free GaN material, which is the first requirement for high-efficiency wire-based optoelectronic devices.

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Research of self-organization of iridium nano-islands for GaN nanowire growth

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Nanometer scale self organized metal islands have a number of applications. They can be used in medicine to kill cancer cells selectively. Another field of application is optoelectronics. Recently lots of studies have been done on improving internal quantum efficiency of nanoscale light emitters: quantum wells and dots, nanowires. At the interface between conductive particles and insulator or semiconductor the excitation of collective electrons, known as surface plasmons, can be observed. Surface plasmons can be coupled to excitons in the active layer of light emitter. At resonant interaction, the exciton emission wavelength coincides with the surface plasmon mode, thus, internal quantum efficiency can be enhanced. Theoretical calculations of light dispersion at iridium-GaN interface showed that surface plasmon resonance lies directly at the band gap of GaN i.e. 3.4 eV. Therefore the iridium (Ir) nano-islands may act same as metal catalyst and surface plasmon resonator. This work aims at research of iridium nano-island morphology primarily attributed to growth of GaN nanowires.

Less than 10 nm, different thickness iridium thin films were deposited on Si (100) substrates by electron beam evaporation. The prepared samples were annealed at 1100°C in hydrogen, nitrogen and oxygen environment. The annealing time varied from 5 min till 60 min. Surface analysis by scanning electron microscopy (SEM) showed that self organizing Ir nano islands of regular shape were formed only when oxygen in the ambient gas mixture during annealing process was included. According to SEM image statistical analysis the surface area size of nano-islands has an exponential distribution with average mean diameter less than 100 nm. The atomic force microscope (AFM) measurements confirmed the formation of nano-islands. The cross-section of nano-islands has been found to be a shape of compressed half sphere as high as 30 nm.

It was expected that silicon dioxide (SiO₂) covering the iridium islands may appear after annealing in oxidizing environment. To confirm that Fourier transformed infrared (FTIR) measurements were carried out in the range of 1000-5000 cm⁻¹. The sharp absorption peak observed at 1100 cm⁻¹ which belongs to Si-O-Si vibrating bonds. Intensity of the peak appeared higher with increased annealing time corresponding to the growth of thicker SiO₂ layer around the Ir nano-islands. The SiO₂ was removed by etching the samples in buffered hydrofluoric HF acid in order to prepare the Ir nano-islands for growth of GaN nanowire.

Photoluminescence polarization in strained GaN/AlGa_xN core/shell nanowire heterostructures

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Conventional c-plane AlGa_xN-based UV-LEDs suffer from the decrease of light output efficiency with increasing Al composition due to high density of dislocations, problems of extraction efficiency and the internal electric field. In particular, it has been shown that for Al_xGa_{1-x}N with $x > 0.12$ the dominant optical transition is polarized parallel to the c-axis, which is detrimental for the emission from the surface. In this context, core/shell GaN/AlGa_xN nanowire (NW) geometry is advantageous to improve microstructural quality, enhance light extraction efficiency and provide more flexibility in the design of the LED active region.

In this work, we report on the optical properties of n-GaN/p-AlGa_xN core-shell NW heterostructure. We show that by strain engineering it is possible to maintain the emission polarization perpendicular to the c-axis in Al_xGa_{1-x}N with x up to 0.20.

The n-type GaN core NWs have been grown by plasma-assisted MBE, followed by epitaxial p-type AlGa_xN shell overgrowth using HVPE technique. The NW structure was studied by SEM/EDX and X-ray diffraction. The core-shell heterostructures consist of two parts: the NW base, about 10 μm long, containing bare GaN core with a diameter of 50-100 nm, and the top, around 7 μm long, where the GaN core is covered by a 500-1000 nm thick Al_xGa_{1-x}N shell with $x = 0.15-0.27$.

The micro-photoluminescence (μ -PL) maps of individual core-shell structures exhibit a set of emission peaks assigned to different NW regions. When exciting the GaN NW uncoated base, a main peak at 3.472 eV corresponds to the donor-bound exciton $D^{\circ}X_A$ in strain-free GaN. The PL spectrum of core-shell part of the NW exhibits two additional features. One, around 3.51 eV, is related to strained GaN, while a second one, between 3.7-3.8 eV, originates from the AlGa_xN shell. The polarization resolved μ -PL analysis shows that the near-band-edge (NBE) peak of the relaxed GaN is polarized perpendicular to the NW axis, according to the selection rules of the Wurtzite crystal. However, NBE from the strained GaN core in the core-shell structure is polarized parallel to the NW axis. Contrary to strain-free Al_xGa_{1-x}N films, the luminescence of the AlGa_xN shell is weakly polarized perpendicular to the c-axis because of the tensile c-axial strain in the shell.

The polarization properties are interpreted by modeling the electronic band structure of GaN and AlGa_xN under uniaxial strain along the c-axis. By means of 6x6 **k.p** analysis, we demonstrate that it is possible to keep the AlGa_xN emission polarized along the c-axis, in order to enhance the light extraction along the NW axis by engineering both the strain and the composition in the AlGa_xN shell.

Electrical and luminescent properties of nanopillar GaN/InGaN MQW LED structures prepared from standard planar structures by dry etching using self-organized Ni nanomask

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Nanopillar GaN/InGaN MQW LED structures with characteristic nanopillar diameter of 100-250 nm and characteristic nanopillar height of about 500 nm were prepared by dry etching of planar structures using a self-organized Ni nanomask. The mask was prepared by deposition of SiO₂ and Ni on top of the structure and annealing. The contacts to these LED nanocolumns were prepared by transferring and annealing graphene monolayers prepared by CVD (four monolayers were used). These nanopillar LEDs showed about two times higher EL intensity compared to their planar opposite numbers. However, C-V and I-V measurements on nanopillar LEDs showed a greater sensitivity of measured parameters to ambient conditions and more pronounced persistent photocapacitance and photoconductivity which is attributed to the impact of defects produced by dry etching in the sidewalls of nanopillars. MCL spectra measurements showed for both planar LEDs and nanopillar LEDs three dominant bands centered near 369 nm, 405 nm and 450 nm, the latter being due to MQW luminescence and the 405 nm band being dominant. Exposure of the samples to the SEM probing electron beam for times between 5 minutes and 40 minutes led to the MCL intensity being steadily repumped from the 405 nm band into the MQW 450 nm band with increasing irradiation time. The amount of the 405 nm band enhancement was considerably higher for the nanopillar sample, suggesting movement and redistribution of lattice defects under the electron beam, more pronounced for nanopillar samples. In order to clarify the nature of the processes involved we also looked at luminescence spectra and electrical properties of undoped n-GaN single layers and nanopillar structures prepared from these layers by dry etching, similarly to the nanopillar LEDs. We observed that the bandedge luminescence of these test structures was lower than for planar test n-GaN layers, most likely due to defects produced by dry etching. Annealing at high temperature in nitrogen and etching in aqueous KOH solution both led to increasing the nanopillar n-GaN bandedge MCL intensity. The results will be discussed in the light of C-V and DLTS measurements on planar and nanopillar n-GaN test structures.

High quality epitaxial r-BN grown by CVD

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The sp^2 -hybridized boron nitride (sp^2 -BN) can adopt two crystal structures; hexagonal (h-BN) and rhombohedral (r-BN) that differ only in stacking sequence. sp^2 -BN is analogous to graphite, but has bandgap of around 6 eV [1]. For the growth of sp^2 -BN thermal CVD is generally used resulting on polycrystalline h-BN or h-BN film containing a large amount of less ordered BN (turbostratic-BN (t-BN) or/and amorphous BN (a-BN) [2]. BN is easily doped to achieve n- or p-type conductivity, making sp^2 -BN interesting for various applications as semiconductor- and optoelectronic material in the UV range. The development of techniques based on sp^2 -BN has been hampered by the great difficulties in growing h-BN and r-BN as an epitaxial film of high material quality. Also the literature is not perfectly clear on the crystalline phase of the films since XRD peaks of h-BN and r-BN overlap [3].

We now demonstrate epitaxial growth of state-of-the-art quality sp^2 -BN and for the first time show that the films are of the rhombohedral phase (r-BN) [4]. We further present a detailed understanding of how the process parameters in the CVD process affect the quality of the r-BN film.

Epitaxial growth of r-BN films on sapphire substrates is achieved in a hot wall chemical vapor deposition reactor using triethyl boron and ammonia as precursors. The influence of the process parameters (temperature, N/B-ratio, B/H₂-ratio and carrier gas composition) on the quality of the grown layers is investigated. X-ray diffraction (XRD) measurements and particularly pole figure measurements reveal that the films are of rhombohedral twinned structure. In addition XRD measurements in Bragg-Brentano configuration show that epitaxial r-BN film can be deposited only in a narrow process parameter window. The 0003 peak at 26.7° of r-BN has a full width at half maximum of only 0.3°, the smallest reported value for sp^2 BN. Outside the optimal process parameters window less ordered BN (t-BN or a-BN) is favored if BN is formed. In addition, a thin strained AlN buffer layer is needed to support epitaxial growth of r-BN film on sapphire whereas a high quality AlN buffer favors turbostratic BN. Pure hydrogen as carrier gas is found to be necessary for the r-BN deposition. The quality of the grown film is affected by the B/H₂ ratio with a decrease of the basal planes spacing with a decrease of boron content in carrier gas. The growth of high quality r-BN is achieved only with low growth rate to avoid possible contamination from impurities as carbon or oxygen atoms. In the view of our experimental data, a chemical reaction scheme for the BN deposition process is suggested.

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Surface morphology of $\text{Al}_{1-x}\text{In}_x\text{N}$ grown by low pressure MOVPE on various templates

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Due to its unique properties the ternary alloy $\text{Al}_{1-x}\text{In}_x\text{N}$ became more and more important in research during the last years. Based on the advantages of $\text{Al}_{1-x}\text{In}_x\text{N}$ compared to $\text{Al}_{1-x}\text{Ga}_x\text{N}$ regarding lattice mismatch and contrast in refractive index to GaN one possible field of application is the replacement of $\text{Al}_{1-x}\text{Ga}_x\text{N}$ cladding layers in current GaN based laser structures. The total strain energy in the device can be reduced this way, whereas the optical confinement can be improved. To minimize optical losses the $\text{Al}_{1-x}\text{In}_x\text{N}$ layers should be of high crystalline quality and very smooth. Their thicknesses have to be in the range of 400 nm to 500 nm. Furthermore there should be no additional defect generation in these layers.

In our experiments we observe that lattice matched, as well as non lattice matched $\text{Al}_{1-x}\text{In}_x\text{N}$ layers grown by low pressure MOVPE on various buffer layers exhibit a typical surface morphology for small layer thicknesses consisting of small features of about 100 nm in diameter and 1 nm in height, some of which are decorated with a pit inside. This behavior of the surface morphology is also well known from literature and stands in contrast to the high crystalline quality of the samples measured by XRD. Rocking curve measurements on the $\text{Al}_{1-x}\text{In}_x\text{N}$ layers reveal values down to 275 arcsecs for (0002) reflex and 580 arcsecs for (10-10) reflex. To investigate the influence of the substrate, the nature of the underlying layer, the strain state of the $\text{Al}_{1-x}\text{In}_x\text{N}$, or the defect density on surface morphology and crystalline quality we performed growth experiments of single layer samples and multi layer structures on various substrates and templates with different layer thicknesses.

We obtained best results for surface morphology from $\text{Al}_{1-x}\text{In}_x\text{N}$ grown on GaN using triethylgallium and ammonia with nitrogen as carrier gas with RMS roughness below 1 nm, but the surface morphology is quite the same for all samples. Only $\text{Al}_{1-x}\text{In}_x\text{N}$ grown on a layer of $\text{Ga}_{1-x}\text{In}_x\text{N}$ shows a different behavior concerning the surface morphology. The surface of these samples consists of two different types of morphologies. On the one hand the surface is made of broad terraces, but on the other hand the grainy morphology is on top. Growing samples with thicknesses around 250 nm revealed that the surface of $\text{Al}_{1-x}\text{In}_x\text{N}$ gets more fine-grained with increasing layer thickness independent of the underlying material. It was not possible to prevent the roughening, but we were able to initiate the fine-grained morphology for thinner layers. The roughening of the surface of thick layers and multi layer structures with a large total layer thickness of $\text{Al}_{1-x}\text{In}_x\text{N}$ mostly comes along with a splitting in composition. The possible causes for the formation of the surface morphology and the splitting in composition are discussed in this contribution.

Composition dependence of critical thicknesses in GaInN/GaN characterized by *in situ* X-ray diffraction measurement

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The GaInN/GaN heterostructure system has aroused wide interest as a promising system for many applications, such as LEDs, laser diodes, sensors, and solar cells. One of the key issues in realizing the high performance devices is to reduce the density of dislocations and growth pits, as they act as nonradiative recombination centers and leakage current paths. The generation of misfit dislocations and growth pits is a well-known phenomenon in heteroepitaxy, where a thin epilayer is grown on a substrate with significantly different lattice parameters. In previous reports, GaInN films with a high InN molar fraction have been described to exhibit growth pits and induce misfit dislocations due to strain relaxation. However, the relaxation sequence remains to be clarified. In this study, we investigate the composition dependence of the critical thicknesses, in GaInN/GaN heterostructures, which were characterized by *in situ* X-ray diffraction (XRD) monitoring grown by metalorganic vapor phase epitaxy (MOVPE).

We evaluated GaInN films on GaN by observing the symmetric (0002) Bragg diffraction using an *in situ* XRD system. An *in situ* X-ray was focused on the sample surface using a Johansson curved crystal mirror, and the diffracted X-ray was detected by a one-dimensional charge-coupled device. *In situ* XRD spectra can be measured similarly to typical XRD spectra in 1 s, the spectra indicate the tilt components of the crystals and the distribution of the lattice constant c . We prepared five sets of samples with InN molar fractions in GaInN of 0.10, 0.13, 0.17, 0.20, and 0.22 by varying the growth temperature and the ratio of the supply gases. We also fabricated and characterized Ga_{0.87}In_{0.13}N and Ga_{0.84}In_{0.16}N films with different thicknesses on GaN. An threading dislocation (TD) density in GaN layer is approximately $3 \times 10^8 \text{ cm}^{-2}$.

From the *in situ* XRD measurements and *ex situ* transmission electron microscopic (TEM) and scanning electron microscopic (SEM) analysis, two strain relaxation features were observed; bent threading dislocations (TDs) with the formation of growth pits, and $a+c$ -type misfit dislocations produced by a $\langle 11-23 \rangle \{ 11-22 \}$ slip system. The *in situ* XRD monitoring system proposed here enabled us to estimate the lattice properties and determine the above strain relaxation features of GaInN films during growth. We denoted the hc_1 and hc_2 for each relaxation points. Here, hc_1 is associated with bent TDs, which form growth pits at the ends of TDs. Moreover, we confirmed that misfit dislocations (MDs) were introduced when the thickness exceeded hc_2 . The critical thickness of hc_1 and hc_2 decreases with increase the InN molar fraction in GaInN. In addition, hc_1 is smaller than the hc_2 , when the InN molar fraction in GaInN is same value. We also compared experimental results and the results of theoretical calculations in refs. [1-4]. The calculation results in ref. 1 comparatively agreed with the value of hc_1 , in this study.

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X-ray multiple diffraction as a new approach to structural characterization of III-nitride layers

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Multiple diffraction effect occurs when two or more lattice points lie on the Ewald sphere at the same moment. Multiple diffraction is measured when a crystal is set in a diffractometer for a primary reflection and then rotated around the diffraction vector (φ -scan). If the primary reflection is forbidden or very weak, multiple diffraction events appear as peaks of “Umweganregung” on the diffraction pattern (Renninger scan). Three wave diffraction combines more the one diffraction geometry, corresponding peaks contain information on different strain components and displacement fields that provides a possibility to obtain the structural parameters using the simplest symmetrical Bragg geometry. Recently it was shown that the multiple diffraction is very suitable for a structural study of the III-nitrides [1-2].

In this paper, multiple diffraction was applied for a detailed study of epitaxial layers GaN, AlN, AlGaN and superlattices on their basis. A series of the layers of different thickness and structural perfection grown on c-sapphire are under study. Renninger scan for the primary forbidden reflection 0001 was used. The three beam diffraction peaks were measured in both φ -scans and θ -scans and their FWHM was analyzed. Main features of the three wave diffraction pattern for heavy distorted layers were ascertained and used for structural characterization of the III-nitride films. From an angular position of the Renninger peaks on φ -scale the lattice parameters a and c of wurtzite structure can be determined. Optimum ways for determination of lattice parameters c and a are suggested. The values of the parameters obtained are used for determination of a composition and strain of the epitaxial layers.

FWHM analysis of the diffraction peaks allows us to control the defect structure of the III-nitride layers. FWHM of the three wave peaks on the scale of azimuth angles (φ -scan) only slightly depends on the dislocation density and a type of the three wave combination. On the other hand a strongly dependence on the structural perfection was observed for the angular width of the θ -scan peaks. The combinations with pure Laue secondary reflections $(1\bar{1}00)/(\bar{1}101)$, $(3\bar{1}20)/(\bar{3}121)$ and $(3\bar{2}10)/(\bar{3}211)$ are most sensitive to the dislocations (most wide peaks are fixed). Most narrow peaks for the samples with the large dislocation density take place for the multiple combination $(01\bar{1}3)/(0\bar{1}1\bar{2})$ with the largest Bragg component of the secondary diffraction vector. For superlattices, it is shown that their parameters can be obtained from diffraction curves of θ - 2θ -mode in forbidden reflection when three wave conditions are satisfied.

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Structural characterization of short period superlattices GaN/AlN

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Short period superlattices (SL) GaN/AlN have been studied by X-ray diffraction methods and Raman spectroscopy. SLs were grown in a horizontal flow reactor at a temperature of 1050° C on c-sapphire substrate using GaN and AlN buffer layers [1]. The period of SLs varied in a range (20 – 60) Å, and the thickness of the structures ranged from 0.3 to 1 µm. X-ray diffraction methods include measurements of the θ –2 θ -mode diffraction curves for symmetrical Bragg reflections, mapping in reciprocal lattice space for asymmetrical Bragg reflections, analysis of broadening of diffraction peaks in different geometry of diffraction, precise measurements of the lattice parameters and determination of the curvature radii. It follows from the average lattice parameters of SLs and the positions of the reciprocal lattice points in the maps that for most of the samples the SLs are fully relaxed as a whole and this state does not depend on the buffer layer material and SL period and thickness. The thickness and strain of individual layers in SL were determined using the diffraction curve simulation and following fit procedure from the θ –2 θ -scans. As a result it was shown that no relaxation between individual layers in SL takes place and the corresponding strain of the layers has the close magnitude about $2 \cdot 10^{-3}$ and the opposite sign (the AlN sublayers are in a tensile state and GaN-sublayers are in compressive one). The strain values obtained were compared with the results of the Raman spectroscopy. The FWHM of the diffraction peaks in different geometry have been analyzed according to approach suggested in [2]. As a result the density of different dislocation ensembles was estimated. It was found that the typical for the III-nitride layers defect structure with the dominating density of the straightforward threading dislocations of screw and edge types takes place for most samples. On the other hand a broadening of the θ –2 θ -peaks in symmetrical Bragg geometry indicates the presence of the horizontal (parallel to surface) dislocations of a density about 10^8 cm⁻². There was not observed any distinct dependence of the dislocation density (in range 10^8 - 10^9 cm⁻²) on the period and total thickness of SLs. However one can conclude that the SLs grown on the two buffer layers (AlN+GaN) are characterized by higher dislocation density.

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Detailed TEM quantifications of threading dislocations in GaN layers grown on Si

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Gallium nitride has reached a great stage of development. Nevertheless, large GaN bulks are still not available, so other semiconductors have to be chosen as substrates of III-N-based devices. Silicon is nowadays the most interesting choice for commercial purposes, due to both its low cost and its well established technology. On the other hand, the use of non-native substrates produces structural defects in GaN, being threading dislocations (TD) the most common in heterostructures with a big lattice misfit (~17% for GaN(0001)/Si(111)). Therefore, finding new ways to reduce the TD densities (TDD) is of great importance for GaN electronics. In consequence, it is fundamental to apply the most reliable methods to measure TDDs in the grown layers.

This work describes, for wurtzite crystalline structures (such as hexagonal GaN of the 2H- polytype), a complete and simple procedure that allows highly reliable TDD measurements. For certain thicknesses and circumstances, the method allowed separating, within a group of dislocations attending to their Burger's vector nature, among their three types (screw (s), edge (e) and mixed (m) TDs). This is based in (i) the combined analysis of planar view (PVTEM) and cross section (XTEM) micrographs; and (ii) the use of commonly-utilized but also of non-conventional reflections to form the images of the dislocations highlighted on a matrix background: $(2\bar{1}11)$ near the $[12\bar{1}3]$ zone axis (~31° away from the [0001] zone axis). In this way, it is possible to obtain micrographs of groups of two or three types of TDs together, depending on the reflections used to get diffraction contrast conditions. Applying these ideas, a statistical TD study is carried out for 3 samples consisting in AlGaIn/GaN-based high electron mobility transistors structures grown on Si (111) under different conditions. Results are analyzed to decide which one of the heterostructures on Si (using an unstrained GaN:C layer (S1), a highly strained GaN:C layer (S2) or an AlN/GaN superlattice (S3)) yields the lowest TDD.

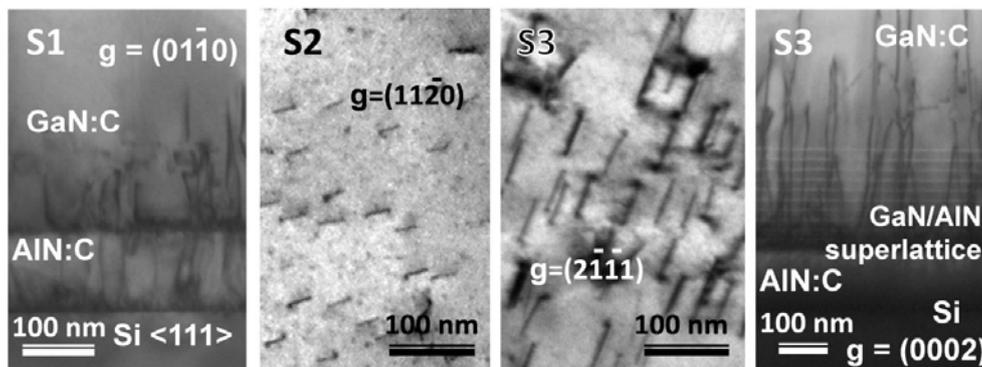


Figure. Diffraction contrast bright field micrographs using many reflections to highlight TDDs. Examples of XTEM of e+m for S1 and S3, PVTEM of e+m in S2 and of e+s+m in S3.

Comprehensive TEM quantification of threading dislocations scaling up through GaN and InN layers grown on sapphire

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Threading dislocations (TD) are the most common structural defects in heterostructures of wurtzite III-N layers mainly due to the lattice mismatch between template and epilayer (i.e. ~14% for Al₂O₃/GaN(0001) and ~11% for GaN/InN(0001)). TDs have a smaller effect on nitride based devices than on other semiconductors in light-emission applications. However, the strategies for reduction of the TD density are valuable for most III-N electronic uses. The role of TDs on GaN in charge carrier recombination is still under debate, and they have been assigned as one main cause of free electrons in unintentionally doped InN films with thickness in the micrometer range. In consequence, it is fundamental to apply the most reliable methods to measure and characterize TDs in epilayers in order to analyze their influence on film properties.

This work extensively analyses TDs at many different heights of both nitride layers in three InN(~1µm)/GaN(~1.5µm)/Al₂O₃(0001) specimens. The difference among them is that the InN films were either unintentionally doped (A), or had an intermediate (B) or high (C) content of incorporated carbon, achieved by supplying gaseous carbon tetrabromide during their plasma-assisted molecular beam epitaxy on MOVPE GaN/sapphire templates. A systematic and reliable quantification of TD densities by transmission electron microscopy allowed drawing some general conclusions. According to their Burger's vectors, the TDs are of screw (s), edge (e) or mixed (m) types. The average ratio of (e+m)-TDs with respect to the (s+m)-TDs in GaN was 6/4, with densities in the range of 10⁹ cm⁻². It was possible to ungroup the dislocations inside the InN film to guarantee that the mixed-TDs are in a negligible content (<10⁸ cm⁻²) compared to the others, having a relative percentage of ~90% of edge type (in the range of 10¹⁰ cm⁻²) with respect to 10% of screw type (~10⁹ cm⁻²), which is in agreement with previous empirical results. Additionally, the density of edge-type threading dislocations decreases with increasing InN thickness, revealing an annihilation rate of ~20% from the InN/GaN interface towards the surface of the 1 µm thick InN film.

Previous experiments in the same samples have revealed that the lattice parameters and good crystalline quality of the InN(0001) films remain unchanged when carbon is intentionally incorporated during the heteroepitaxial growth of InN on GaN, while the electron concentration linearly increased with higher carbon contents. However, the formation of surface pits with higher densities as carbon increased was observed, in addition to monotonic changes of electron transport, optical, and surface electronic characteristics. Since the present TEM study demonstrates that the density of TDs remain in similar numbers for the three samples in both the GaN and the InN layers, in agreement with XRD observations establishing that the crystal quality of InN is not influenced with increasing carbon content, it turns out that the main reason for the cumulative n-type doping, and of the slight shift of the valence band maximum, is the presence of carbon impurities rather than TDs that may enhance to the electron concentration in a constant manner.

Semipolar gallium nitride layers on Si grown by HVPE: TEM investigation

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GaN is a promising material for high-performance green and ultraviolet light-emitting devices (blue, green, and white light emitting diodes and blue laser diodes). But the presence of electrostatic fields generated by the spontaneous and piezoelectric polarization along the [0001] axis in wurzite GaN layers reduces the luminous efficiency of the devices. The zinc blende phase of nitride semiconductors is not piezoelectric when grown along [001]. Unfortunately this phase is thermodynamic metastable. Another way of field reduction is to use wurzite gallium nitride grown in semipolar direction (semipolar GaN) [1,2]. In recent years, much attention has been given to the growth of GaN on Si substrates. This interest is related, first, to the prospects for integration of the gallium-nitride and silicon electronics technologies and, second, to the possibility of using inexpensive single crystal silicon substrates, available in large diameters (up to 200 mm). Furthermore Si substrate has reasonable thermal and electrical conductivity. Thus growth of semipolar ($\bar{1}\bar{1}01$)GaN has been attempted on a patterned (001) silicon substrate adopting selective area MOVPE [3]. The growth was initiated on (111) facets of the Si, which had been prepared by anisotropy etching.

In this report results of TEM investigation of GaN layer structure grown by hydride-chloride vapor-phase epitaxy (HVPE) on silicon substrate without any prior etching are presented. It is necessary to emphasize, that method HVPE allows to grow thick (about 10 μm) layers. A 50-nm-thick buffer layer of silicon carbide on Si(001) substrate was formed using an original solid-phase epitaxy method [4]. It is established, that GaN consist of oriented grains several hundred nanometers in size. The majority of grains has wurzite structure, {0001} GaN planes are oriented parallel to {111} Si planes, so that the deviation of a layer orientation from polar one made about 55°. Also there was revealed the cubic modification of gallium nitride in the layer.

Thus, in the first time, semipolar GaN layer has been grown by HVPE on Si(001) substrate.

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Comparison of the crystalline quality of GaN films on a- and c-sapphire substrates using low-temperature AlN buffer layers

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Light-emitting diodes (LEDs) based on GaN with a sapphire substrate have been utilized in advanced commercial applications such as the backlight in liquid crystal displays. However, the dislocation density of over 10^8 /cm² in GaN films impedes further increases in the output power of LEDs. In general, a- and c-sapphire are used for the growth of c-plane GaN. The crystalline quality of GaN films on a-sapphire is reported to be better than that of GaN films grown on c-sapphire [1]. We believe that understanding the growth mechanism of GaN on a- and c-sapphire will lead to the improved crystalline quality of GaN films. In this study, to clarify the difference in the crystalline quality of GaN films on a- and c-sapphire, the low-temperature (LT) AlN buffer and the initial GaN growth on a- and c-sapphire are investigated and compared.

The samples were grown on a- and c-sapphire substrates by metalorganic chemical vapor deposition. An LT-AlN buffer layer was deposited at a temperature below 500°C. The LT-AlN buffer layer was annealed under a NH₃ and H₂ mixture while ramping up the substrate temperature to above 1000°C. Then, a GaN film was grown above 1000°C. Samples of as-deposited and annealed LT-AlN buffer layers as well as GaN films with thicknesses of 400, 800, and 1600Å were prepared to investigate the growth mode of GaN films on a- and c-sapphire.

The surface morphology of the LT-AlN buffer layers was characterized by atomic force microscopy (AFM). The surface morphologies of the as-deposited LT-AlN buffer layers on a- and c-sapphire were almost the same. After annealing, the surface morphology of LT-AlN on both a- and c-sapphire changed to an islandlike structure. However, the AlN islands on c-sapphire were smaller than those on a-sapphire. The selected-area electron diffraction pattern from a (0001)AlN plan-view transmission electron microscopy specimen showed that the diffraction of the AlN (10-10) spot on c-sapphire is broader than that on a-sapphire. AFM images of the surface of the GaN films with thicknesses of 400, 800, and 1600Å on a- and c-sapphire showed that the size of the GaN islands on both a- and c-sapphire becomes large as the growth advances. However, the GaN islands on a-sapphire are larger than those on c-sapphire. The size of the GaN islands is correlated with the size of the annealed AlN islands. To investigate the relationship between GaN island density and dislocation density (DD), DD was calculated using the equation $DD=F^2/(9b^2)$ [F: full width a half maximum (radians) of the (10-10)GaN x-ray rocking curve, b: Burger's vector]. It was found that DD is correlated with the GaN island density, that is, the lower the GaN island density, the lower the DD. These results indicate that decreasing the island density of the annealed AlN buffer layer is essential for increasing the crystalline quality of GaN films. It is therefore important to control the solid-phase epitaxy of AlN buffer layers by annealing.

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Evaluation of Metastable-Phase Inclusion in PR-MOVPE-Grown InN Films by EBSD

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Among III-V nitrides, InN has a smallest bandgap of about 0.7 eV. Because of this small bandgap, InN has been regarded as a promising material for optoelectronic devices in the optical communications systems. However, the improvement of the crystal quality is difficult due to a high nitrogen equilibrium vapor pressure of InN. To overcome this issue, we have developed a pressurized-reactor metalorganic vapor phase epitaxy (PR-MOVPE). However, the inclusion of the metastable zincblende (ZB) phase into the stable wurtzite (WZ) phase was observed in some of the samples, which was detected by the Raman scattering and X-ray diffraction (XRD) pole figure measurements. Especially XRD pole figure revealed that the ZB phase is included as twinned crystals [1]. The effect of phase purity on dislocation density was also studied by XRD rocking curve measurements, and structural model of ZB inclusion was proposed [2]. However, the actual distribution of ZB phases was still unknown, since the XRD measurement cannot identify the microstructure. In this paper, we report the microstructure of ZB inclusion in PR-MOVPE grown InN, as well as its temperature dependence, which were elucidated by the orientational analysis using electron backscatter diffraction measurement in a scanning electron microscope (SEM-EBSD).

InN films were grown directly on nitrided *c*-plane sapphire substrate at 1600 Torr by PR-MOVPE. Growths were performed for 4 hours after the thermal cleaning and nitridation. Trimethylindium flow rate was set to 15 $\mu\text{mol}/\text{min}$, and NH_3 flow rate was set to 0.38 mol/min. Growth temperature (T_g) was varied from 500 to 700°C with 25°C intervals. Thicknesses of the films were 100~200 nm. EBSD measurements were performed using SEM (JEOL, JSM-7001FA) equipped with EBSD system composed of fluorescence screen and CCD camera.

Orientation maps of ZB-InN obtained by EBSD showed that there is significant inclusion of ZB-InN at T_g lower than 600°C, and the enlargement of domains has been observed with increasing temperature. This enlargement may be the result from the increase in the adatom surface migration with increasing the growth temperature. Results also clearly show the coexistence of ZB-twinned-domains at the surface. This result supports the structural model given by previous report [2], which proposed the coexistence of ZB domains which were located side-by-side with WZ domains and twinned ZB counterparts. Since this model shows that the edge dislocations are inevitably formed at the boundary between WZ domains and twinned ZB domains, when T_g are increased, not only the decrease of the amount of inclusion of ZB-InN but also the increase of domain size may reduce the density of edge threading dislocation. The result of T_g dependence of the WZ phase purity measured by EBSD closely agreed with the result of XRD pole figure. However, EBSD detected ZB-InN for T_g 575°C, while XRD pole figure did not detect it. This may be because the size of the individual ZB-InN domains was too small to detect by XRD in this intermediate temperature.

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Structural Study on Nitrided C-sapphire Substrate by Grazing Incidence X-ray Diffraction and Transmission Electron Microscopy

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Nitridation treatment for GaN growth is the critical step for the defect reduction, high electron mobility and yellow luminescence reduction in the subsequently deposited films. Keller *et al.* reported that the metalorganic chemical vapor deposition (MOCVD) grown GaN on the longer nitridation of sapphire at 1050°C showed two order of higher dislocation density, yellow band luminescence, low hall mobility, and high carrier concentration than the GaN film with the shorter nitridation [1,2]. In order to understand the nitridation effect, first of all, it is highly needed to find out the overall chemical compounds formed on the nitrided sapphire surface and each crystalline orientation relationship. Although X-ray diffraction (XRD) method is generally used to find out them, it is very hard to detect any peak by conventional XRD because the thickness of the nitrided layer is normally below several nanometers. In this presentation, in order to investigate crystallographic and structural properties of the nitrided sapphire substrates, a grazing incidence X-ray diffraction with a synchrotron radiation and cross-sectional high-resolution transmission electron microscopy were performed on the sapphire surface nitrided at 1080°C for 30min. The thickness of the nitrided layer was about 2 nm. It was found out that the w-AlN, non-rotated zb-AlN and 30° rotated zb-AlN were formed on the nitride sapphire surface. Considering the atomic configurations, the 30° rotated zb-AlN shall form the in-coherent interface and has higher activation energy of formation, while the non-rotated zb-AlN shall form the coherent interface. Therefore, the non-rotated zb-AlN shall be formed preferably compared to the 30° rotated zb-AlN [3].

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A Study on the Heteroepitaxy GaN Growth on Various Powders by Hydride Vapor Phase Epitaxy

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Recently, metallic buffers like TiN, ZrB₂, HfN and ScN have been tested for application to high quality GaN on Si because of relatively low lattice mismatches [1-4]. Silicon substrate has many benefits, such as cheapness, large area, and conductivity [4]. Although much research has been done to find a new buffer material, AlN and TiN have also been used as buffer layers for GaN growth on Si substrate. In reality, the evaluation process for finding new buffer materials requires considerable effort. We investigated the nucleation and growth behavior of GaN on various powders by hydride vapor phase epitaxy. In relative comparison, the nucleation tendency of GaN on each powder can be summarized as AlN > LaN, TiN, NbN > ZrN > ZrB₂ > VN, BeO, indicating that the number of nucleation sites increased from right to left. It is well known that AlN and GaN compounds are intermixed by a consolute above 305K [5]. LaN and NbN have not yet been reported as buffer materials for GaN growth. Of these, NbN is expected to be a good buffer material because the interatomic distance on the NbN (111) plane has only 2% difference from that on the GaN (0001) plane. In order to understand this tendency for differential GaN nucleation at various hetero-interfaces, we must consider the many factors of facet surface, surface atoms, polarity and strain energy due to lattice misfit, and so on. A few studies have been performed on the interfacial energy calculation between the GaN (0001) plane and the ZrB₂ (0001) or CrN (111) plane [2,6,7]. Despite these studies, it seems to be difficult to understand the nucleation tendency of GaN because it is necessary to consider more complex factors of the intermediate phase and its kinetics. Our experimental approach is expected to yield a simple route for the screening of various candidate materials in terms of GaN nucleation.

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Annealing effect for unification of in-plane domain in AlN layers grown from Ga-Al flux

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AlN is a promising substrate material for AlGaIn-based ultraviolet light emitting diodes (UV-LED), because of its high thermal conductivity, high ultraviolet transmittance and small mismatch with AlGaIn. Recently, the authors have investigated a novel liquid phase epitaxy (LPE) technique using Ga-Al flux under normal pressure of nitrogen. In this technique, surface nitrided sapphire substrate [1, 2] was used as a template to achieve homoepitaxially growth. We have succeeded to grow 1.2- μm -thick AlN layer on the nitrided sapphire layer for 5 h at 1573 K from Ga-40mol%Al flux [3]. The grown AlN layer has a high c-axis orientation. The full width at half maximum (FWHM) value of (0002) x-ray rocking curve (XRC) was 50 arcsec. However, the XRC of AlN (10-12) exhibits a split peak with 1-deg difference, which indicates the existence of rotational domains in-plane. The rotational domains originated from the nitrided layer of c-plane sapphire used as a template.

In this study, we have attempted annealing of the nitrided substrates before the LPE growth to unify the rotational domain. The nitrided c-plane sapphire substrates were kept for 2 h at temperatures of 1173, 1373 and 1573 K under nitrogen atmosphere. After that, the substrates were immersed in the flux, and nitrogen gas was injected into the flux. Subsequently, the substrates were heated up to the growth temperature, 1573 K, and were kept in the flux for 5 h.

The existence of rotational domains was evaluated from split AlN (10-12) XRCs. From the comparison of XRCs of AlN grown at each temperature, the intensity at higher-angle peak decreased with increasing temperature. On the other hand, the intensity at lower-angle peak increased with increasing temperature. Thus, the annealing process before LPE is significantly effective for unification of the rotational domains.

We consider that the origin of the rotational domain is caused by the distortion of oxygen ions arrangement in sapphire lattice [4]. In c-plane oxygen layer, two kinds of distances between adjacent oxygen ions exist. The rotational domains in the nitrided sapphire may be caused by this distortion. During the annealing process at 1573 K, amplitude of lattice vibration of the sapphire became larger. This lattice vibration makes the difference between two kinds of distances of oxygen ions obscure, therefore, rotated domains in the nitrided sapphire was unified during the LPE process. The distances of oxygen ions are 0.252 nm and 0.287 nm *i. e.*, distortion of oxygen per an ion is 0.017 nm. The mean-square displacement of oxygen lattice vibration was estimated as 0.012-0.013 nm at 1573 K, which reaches over 70 percent of the oxygen distortion. Thus, the thermal lattice vibration greatly contributes the unification of the rotational domains for the LPE process.

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Nano-layers of nitride semiconductors grown by MEAGlow epitaxial technology and their low-dimensional optical properties

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Both GaN and InN epitaxial nano-layers are grown on sapphire substrates (0001) by novel plasma based migration enhanced afterglow MOCVD (MEAGlow) epitaxial technology [1,2]. The layers are grown at the Semiconductor Research Laboratory, Lakehead University. For this technique metal atoms (Ga and In respectively) are deposited on c-axis oriented sapphire substrates at certain relatively low temperatures. The metal layers are subsequently nitrided by nitrogen plasma. In some cases several cycles of metal deposition and subsequent plasma nitridation are used. The films have been thoroughly characterized by atomic force microscopy and extremely smooth surfaces with very low RMS surface roughness are found. The low RMS surface roughness of the Atomic Force Microscopy data, in conjunction with other measurements (in particular X-ray diffraction data), indicates that good quality single crystals GaN and InN respectively are observed over the scanning areas of the probes. For instance, typical AFM topography images show RMS surface roughness of lower than 1 nm for scan areas of $2 \times 2 \mu\text{m}^2$, with some samples showing RMS surface roughness values of less than 0.2 nm over limited areas. Also study of low-dimensional optical properties of these epitaxial nano-layers (of GaN and of InN grown on sapphire substrates (0001)) is presented. Experimental results are obtained for several of samples and they show variations of the optical absorption edges in range 1.4 – 3.9 eV for GaN and 0.6 – 2.8 eV for InN, which are different in comparison with the known energy band gaps of GaN and of InN respectively. Theoretical investigations of these unusual variations of the absorption edges are done by using Linear Combination of Atomic Orbitals (LCAO) electron band structure calculations. Variations of the lattice constants of the first deposited atomic layers of GaN and of InN are found due to influence of the sapphire substrate. It determines defects in the structures of GaN and of InN and the corresponding LCAO matrix elements are found. The LCAO electron band structures on nano-level are calculated in consideration of interactions between nearest-neighbour orbitals. Electron energy pockets are found in both the conduction and the valence bands at points Γ of the electron band structures. Also it is found that these pockets are on distances, for which there are overlaps between electron wave functions describing localized states belonging to the pockets, and as result tunnel optical absorption [3] has place. This type of absorption determines the variations of the optical absorption edges. Also the calculated LCAO electron band structures show existence of excitons of the structure [3] in these layers of GaN and of InN. Both the binding energies and the first hydrogen like energy levels of the excitons of the structure are determined. It is found that annihilations between the electrons and the holes belonging to the excitons of the structure determine significant “blue shift” of the peak of the PL spectra for these samples.

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Migration Enhanced Afterglow Growth of GaN at Low Temperatures

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Migration Enhanced Afterglow (MEAglow) is a new epitaxial film growth technique for compound semiconductors. The technique utilizes a large area scalable hollow cathode plasma source described elsewhere [1]. Recent advances in the technique have allowed film growth rates to increase from < 20 nm/hour in early 2011, to over 450 nm/hour in early 2012 for film growth temperatures of less than 650° C. Ga-face GaN grown directly on nitrided sapphire substrates has been extended down to film growth temperatures of 500° C, with molecular terraces are visible for films grown down to 530° C. A working GaN LED grown at less than 560° C has recently been demonstrated. Clearly the MEAglow technique is demonstrating excellent material properties for GaN grown at exceptionally low temperatures.

We will provide details of some of these recent advances including SEM, AFM, XRD and some LED characterization results.

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Growth of thick AlN and GaN layers with atomically smooth and droplets-free surface by PA MBE under strongly metal rich conditions

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III-Nitride layers with atomically smooth surface are commonly grown by PA MBE under Me-rich stoichiometric conditions ($F_{III}/F_{N^*}>1$). This enables one to increase the low surface mobilities of both group-III and nitrogen adatoms at the relatively low growth temperatures $T_S\sim 700-800^\circ\text{C}$ used in this technology. However, accumulation of excess metal on the epitaxial surface leads to formation of the metal microdroplets on the surface that complicates devices manufacturing due to the short-circuit problem and nonplanar surface morphology [1]. This paper reports on PA MBE of AlN and GaN layers with atomically smooth and droplet-free surface morphology of different thickness (up to 3 μm) under the Me-rich stoichiometric conditions by using continuous activated nitrogen flux and periodically interrupted Me flux, with the duration of interruptions being precisely controlled by the laser reflectometry (LR).

Both AlN and GaN(0001) thick layers were grown by PA MBE on AlN/c-Al₂O₃ buffer layers with the growth rate of 0.5 $\mu\text{m}/\text{h}$ determined by continuous activated nitrogen flux only at the strongly Me-rich conditions ($F_{III}/F_{N^*}=1.2-2.0$) and $T_S=650-840^\circ\text{C}$. Both continuous and pulse group-III atom flux modes were studied. In the latter, the time duration before closing the group-III atom source (so-called metal accumulation stage) was varied within the range of 60-360 s. The surface morphology and growth rate were monitored by RHEED and LR (532 nm). The substrate temperature was measured by an infra-red pyrometer with an accuracy of 10 $^\circ\text{C}$. The surface morphology was evaluated by using atomic force, scanning electron and optical microscopes.

The continuous growth of 1- μm -thick III-N films at $F_{III}/F_{N^*}\geq 1.2$ and relatively low substrate temperatures ($<700^\circ\text{C}$ and $<790^\circ\text{C}$ for GaN and AlN, respectively) leads to similar droplets formation with a density of $\sim 10^4\text{ cm}^{-2}$, a characteristic diameter $\sim 10\text{ }\mu\text{m}$ and a height $\sim 2\text{ }\mu\text{m}$. In the case of the pulse mode, the growth of III-N films continued during 30-180 seconds after closing of the group-III atom sources, as revealed by LR. Dependences of the characteristic time duration (excess metal consumption stage) on the substrate temperatures and group-III atom flux intensities were used to elucidate the kinetic parameters of metal adsorption/desorption processes. Solution of a set of flux balance equations made it possible to evaluate both the desorption rate of metal atoms and the excessive metal volume accumulated on growth surface during the accumulation stage. The former demonstrate the Arrhenius type temperature dependences with activation energies close to the values for free metal evaporation in vacuum ($2.5\pm 0.3\text{ eV}$ for Ga on GaN and $3.0\pm 0.3\text{ eV}$ for Al on AlN). Moreover, this pulsed technique allowed growth of thick III-N films at the strongly metal rich conditions with the growth rate as high as in the continuous mode ($\sim 0.5\text{ }\mu\text{m}/\text{h}$) without metal droplets.

Thus, the III-N films with a thickness up to 3 μm and atomically smooth droplet-free surface morphology (with $\text{RMS}<0.43\text{ nm}$ over $2\times 2\text{ }\mu\text{m}^2$ area) have been grown by the developed technique.

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GaN electron spectra and lattice structural parameters under elastic strains

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The use of gallium nitride (GaN) in opto- and microelectronics devices in the form of epitaxial films on the heterogeneous substrates - Al₂O₃, SiC, Si, Si, etc., generates in the structures the elastic strains of compression/expansion that modifies the lattice structural parameters, electronic spectra of GaN material and affects the characteristics of the devices. Thus, in GaN films on Al₂O₃ substrate the elastic deformation ϵ_a and ϵ_c may be about $-1.5 \cdot 10^{-3}$ и $7.3 \cdot 10^{-4}$ values respectively, which corresponds to the value of the elastic stress at about - 0.7 GPa in the basal plane of GaN. Therefore, the studies of the influence of elastic stress on the lattice structural parameters and electronic spectra of GaN attract the considerable interest.

First principle calculations are carried out to estimate the structural stability and the influence of the elastic compression/expansion stress on the lattice structural parameters, the electronic spectra of material and the energy position of the charge-neutrality level (CNL) in GaN when exposed to external hydrostatic compression and the compression/expansion strain in the direction of the hexagonal *c* - axis and biaxial compression/expansion strains in the basal plane (0001) of the crystal. Stability of the crystal-chemical phases *zb*, *wz* and *rs* and the changes of the lattice structural parameters (*a*, *c*, *c/a*, *u*) of gallium nitride vs the hydrostatic compression and the critical elastic pressure phase transition values $P_t(wz \rightarrow rs) = 44.0$ GPa and $P_t(zb \rightarrow rs) = 43.1$ GPa are estimated. The experimental values P_t , estimated from the X-ray diffraction experiments and Raman measurements data give (42-47) GPa values for $P_t(wz \rightarrow rs)$ transition. Appreciated the change of the energy gaps in the high symmetry points Γ , *A*, *L*, *K*, *M*, *H* in the electron spectrum of GaN, as well as the CNL energy position change upon the hydrostatic compression, single-axial and biaxial compression/expansion strains and the pressure factors for these phenomenon are relevant. As at the case of the hydrostatic compression, at the anisotropic strains is the opposite changing of *u* - parameter in relation to character of $\gamma = c/a$ parameter deformation owing to the resistance of the Ga-N chemical bond to the external elastic deformation influences and determines the *u* - parameter reduction as the lattice parameter *c* increases. Elastic compression along the *c* - axis reduces the valence angles and convergence of anionic and cationic planes to the hexagonal structure known as unbuckled wurzite (*hx*) structure. The critical phase transition (*wz* \rightarrow *hx*) corresponds to single - axial strain at about -31 GPa.

The experimental data on the lattice parameters *a* and *c* modification upon the heavily reactor neutrons bombardment of the epitaxial GaN films on sapphire substrate are presented.

Homogeneous epitaxial InN films on the two-inch Al₂O₃ substrates grown by LP- MOCVD method

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Last years InN is considered to be a very attractive material for future photonic and electronic devices owing to its outstanding material properties like smallest effective mass, largest mobility, highest peak and saturation velocities, and smallest direct band gap in nitride semiconductors. Research of InN film growth on industrially interesting two-inch (0001) and (11-20) Al₂O₃ substrates by low pressure metal-organic-chemical-vapor-deposition(LP-MOCVD) method is carried out at this work. Growth processes were conducted on MOCVD system with the vertical reactor and the induction heating of substrates. TMI_n and NH₃ were used as source materials. Growth pressure was 300 Torr and V/III ratio reached values $2,6 \times 10^6$. Growth was conducted in a nitrogen stream. The big lattice-mismatch of sapphire and InN (17 %) does necessary use of buffer GaN layer. Its growth was carried out by a two-step growth process. At first the thin nucleation GaN layer was grown at temperature 550°C. This layer was annealed at 1100°C for 10 min and then GaN buffer layer 1 micron thickness was grown at 1100°C also. After reception of a buffer layer the film InN has been grown up on it at temperature between 500 and 550°C. All films were grown to a thickness of 300 nm. The growth rate of the InN film grown was about 200 nm/h. Comparative study of epitaxial InN films was performed with use of optical and electronic microscopy, X-Ray diffractometry, secondary ion mass spectrometer, photoluminescences, and also, probe methods. The grown buffer on two-inch substrates had excellent morphology of its surface. The full width at half maximum (FWHM) of rocking curve for buffer GaN layer did not exceed 0,2°. Obtaining of mirror smooth homogeneous buffer layers has allowed to provide excellent uniformity of InN films on the area of a two-inch substrate. InN films had mirror smooth surface and good structural perfection. SIMS analysis of InN/GaN/Al₂O₃ structures has shown presence of homogeneous InN film and also InGaN area between buffer GaN layer and epitaxial InN film. The as-grown materials were of n-type, with electron concentrations in the range $(1-2) \times 10^{19} \text{ cm}^{-3}$ and Hall mobilities of about 500 cm²/V×s at room temperature. The intensive photoluminescence was observed for InN films at a room temperature with position of peak at 0,77 eV. Conditions allowing to provide high uniformity of photoluminescence parameters were found. Study of a the map of a photoluminescence of the received InN films by RPM-2000 system have been performed. Four parameters were applied for description of a spectrum of a photoluminescence: wavelength in the maximum of a spectrum, intensity at the maximum, integral intensity and a value of FWHM for photoluminescent peak. Dispersion of this parameter are 1.5, 5.4, 4.6 and 3.3 %, respectively.

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The growth and characterization of In rich InGaN solar cell epitaxial structures by MOCVD

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Solar energy is widely studied as an endless, reliable and safe energy source. It is important to increase the photovoltaic (PV) conversion efficiency of the solar cell devices. The highest efficiency (~43 %) was achieved by GaAs based multiple junction solar cells. In order to increase the efficiency of solar cell wide band gap material such as III- nitrides can be another future alternative material system.

InGaN is a promising ternary material that the band gap can be tuned in a range corresponding to wavelength from 0,7eV (InN) to 3,4 eV (GaN). Tunable bandgap (0.7 eV to 3,5 eV) and higher absorption coefficient (approximately 10^5 cm^{-1}) makes $\text{In}_x\text{Ga}_{1-x}\text{N}$ alloy a potential candidate for high efficiency photovoltaic application. However on the other hand it is difficult to maintain the crystal quality while growing high In composition InGaN layer due to fundamental thermodynamical limitations and difference in crystal constants.

In this paper we present ~ 200nm thick $\text{In}_x\text{Ga}_{1-x}\text{N}$, for wide range of Indium composition (x: 0.2 to 1.0) layers grown by MOCVD (Metal Organic Chemical Vapor Deposition). XRD, AFM, Hall and etc. characterization results as well as basic Solar Cell design according to high quality InGaN layers will be demonstrated.

Influence of the V/III ratio on quality of thick epitaxial AlN layers grown on sapphire by High Temperature Hydride Vapor Phase Epitaxy (HT-HVPE)

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Thick epitaxial AlN layers were grown on (0001) sapphire using High Temperature Hydride Vapor Phase Epitaxy (HT-HVPE). The experimental set-up consists of a vertical cold-wall quartz reactor working at low pressure (10 torr). The sapphire substrate is placed on a graphite susceptor heated by induction at 1500°C. The reactants used are ammonia (NH₃) and aluminum chlorides (AlCl_x) species *in situ* formed via Cl₂ reaction with high purity aluminum pellets.

As-grown AlN layers have been characterized by Scanning Electron Microscopy (SEM) and X-ray diffraction rocking curves (XRC).

The influence of the V/III ratio on the surface morphology and crystalline state and quality at constant growth-rate and constant layer thickness is presented. Typical growth rates of around 5 μm.h⁻¹ with a total thickness of 5 μm have been chosen for such thick epitaxial AlN layers.

A kinetic and mass transfer analytical model, explained in a previous paper [1], has been used to estimate the experimental conditions in order to keep the growth rate constant while varying the V/III ratio.

This set of experiment is an extension of a previous work of Claudel [2] based on the study of the V/III ratio effect on the AlN epitaxial growth on SiC and AlN templates for lower temperature 1400°C and higher growth rate from 10 to 14 μm/h. First results obtained seem to show that a higher V/III ratio could be the optimal condition for AlN growth on sapphire substrates (compared to the V/III value of 1.5 previously obtained on AlN template).

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- [2] A.Claudel, E. Blanquet, D. Chaussende, R. Boichot, B. Doisneau, G. Berthomé, A. Crisci, H. Mank, C. Moisson, D. Pique, M. Pons, *Journal of Crystal Growth* **335**, 17 (2011)

Influence of the V/III ratio in the gas phase on thin epitaxial AlN layers grown on (0001) sapphire by High Temperature Hydride Vapor Phase Epitaxy (HT-HVPE)

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Since 2007, AlN epitaxial growth on (0001) sapphire (α -Al₂O₃) substrates has been investigated at high deposition temperature above 1200°C.

Previously, our group has reported the growth of thick epitaxial AlN layers by High Temperature-Hydride Vapor Phase Epitaxy (HT-HTVPE) on both silicon carbide (SiC) and sapphire substrates from aluminum chlorides and ammonia at high temperature, up to 1600°C [1]. However, due to high growth rates (>20 $\mu\text{m}/\text{h}$) and etching phenomenon at high temperature, direct AlN heteroepitaxial growth with good crystalline quality is complicated to stabilize.

In this study, NH₃ (g), HCl (g) and solid Al were used as source materials to grow AlN layers. In order to study lower growth rate (< 5 $\mu\text{m}/\text{h}$) at high temperature ($T > 1200^\circ\text{C}$), we have studied the growth of thin (450 nm-thick) epitaxial AlN layers at 1500°C. Such layers, showing good crystalline quality and well oriented along c-axis, could be used as a substrate preparation for a further epitaxial growth at higher temperature ($T > 1500^\circ\text{C}$) and higher growth rate (> 20 $\mu\text{m}\cdot\text{h}^{-1}$).

As-grown thin AlN layers have been characterized by Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM), X-ray diffraction rocking curves (XRC), Transmission Electron Microscopy (TEM), and Raman spectroscopy.

The influence of the N/Al ratio in the gas phase on growth rate, surface morphology, roughness, crystalline state and quality is presented. In particular, an optimum for surface roughness and crystal quality (deduced from X-ray rocking curve FWHM on symmetric and asymmetric reflections) is obtained for N/Al ratio around 7.5.

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Understanding and controlling In incorporation on InGaN surfaces: An ab initio approach

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The growth of high quality and high In content InGaN alloys is challenging: Low growth temperatures are required to achieve high In-content, reducing effects such as In segregation and spinoidal decomposition, whilst higher temperatures are preferred to obtain high crystal quality. Although the {0001} (Ga-polar) surface is so far the technologically most relevant growth surface, recent experimental results have provided evidence that greater In-incorporation can be achieved instead by the {000-1} (N-polar) surface.

A first step in achieving full control on the growth and properties of N-polar InGaN surfaces is to gather a fundamental understanding of the relevant atomistic surface processes as well as to identify and investigate the differences in the growth of the N-polar face compared to the Ga-polar face. In this work, the incorporation of In into N- and Ga-polar surfaces is investigated using density functional theory (DFT) calculations. Total energies for an extensive range of reconstructions on Ga-polar and N-polar surfaces are calculated and surface phase diagrams are constructed showing the most stable structures as a function of growth conditions. A substantially stronger binding for In adlayer/s and for In adlayer/s with sub-surface In is found for N-polar compared to Ga-polar. Kinetic DFT calculations are also performed to address the competing effect of In segregation, with the surface segregation effect found to be weakest for the N-polar surface. Based on these results, the mechanisms of In incorporation, In segregation and also the self surfactant effect will be discussed in detail.

Based on our results we will discuss the mechanisms of In incorporation and In self surfactant effects and we will elucidate the interplay of strain vs. bond enthalpies on In surface segregation. Finally, the effect of surface polarity on In incorporation and surface segregation will be discussed in detail.
We gratefully acknowledge the EU for funding.

HVPE of Al_{0.45}Ga_{0.55}N layers on trench patterned sapphire

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In the last years the fabrication of AlGa_N based UV-LEDs attracted increasing attention, not at least due to the potential to replace mercury vapor lamps in a wide range of applications. Unfortunately, the efficiency of UV LEDs is highly affected by dislocations due to the heteroepitaxial growth of the device structure. A substrate that reduces the lattice mismatch and subsequently the dislocation density will enhance the device efficiency. For LEDs emitting in the UV-B spectral range high quality AlGa_N in a medium composition range would fulfill these requirements. It has already been shown that AlN and GaN substrate material can be fabricated by hydride vapor phase epitaxy (HVPE). We have developed the growth of AlGa_N layers with growth rates of about 30 μm/h by HVPE, which have the potential to serve as pseudo substrates.

For AlGa_N layers grown planar on c-plane sapphire strain induced distortions like cracking and polycrystalline growth were found to degrade the layer quality and limit the layer thickness. Hence, strain-relief by epitaxial lateral overgrowth (ELOG) on a trench patterned sapphire substrate was investigated. The trenches were prepared in [11-20]_{sapphire} direction exhibiting a trench width of 4 μm, a trench depth of 4.5 μm and a ridge width of 6 μm.

We investigated the growth of 5 μm Al_xGa_{1-x}N with x=0.45±0.05. At a total pressure of 800 hPa the AlGa_N was deposited on an in-situ grown AlN buffer layer of 500 nm thickness. The overgrowth of the patterned sapphire resulted in a saw tooth like surface structure. Cross section scanning electron microscopy images show a correlation of this surface structure to fast growth starting at the sidewalls of the trenches. From x-ray diffraction measurements it can be concluded that in addition to the (0001) AlGa_N growth on the ridges (11-22) AlGa_N is deposited on the m-plane oriented sidewalls of the trenches. This semipolar material intersects the c-plane grown material from the ridges and forms the saw tooth like surface structure after coalescence. From literature the growth velocity of different Ga_N crystal facets is known to depend on the total pressure during growth. Hence, to find a way to favour the c-plane AlGa_N growth on the ridges over the semipolar sidewall growth we investigated the influence of the total pressure. Best results were achieved at a reactor pressure of 400 hPa at which the c-plane growth from the ridges dominates over the sidewall growth. In this case, the c-plane orientation of the AlGa_N layer prevails. The use of the patterned sapphire substrate and a total pressure of 400 hPa prevented surface distortions like cracking and polycrystalline growth. The FWHM of the XRD ω-rocking curve of the 002 reflection is 1450 arcsec and the one of the 302 reflection is 2030 arcsec. The surface morphology of the AlGa_N layers has been improved by optimization of the V/III ratio and the miscut orientation of the sapphire substrate. Structural and optical properties of these AlGa_N layers will be presented.

Top-side satellite to satellite temperature control in planetary MOCVD reactors using a novel gas-foil-rotation based temperature controller

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The control of the effective surface temperature of the wafer is of paramount importance for the production of InGaN-based light emitting diodes (LED), especially if automation of wafer and process carrier (satellite) exchange is taken into account. Slight changes in satellite properties, such as weight, surface finish or wear during use can cause the surface temperature to differ from satellite to satellite and run to run.

In Planetary Reactors the thermal coupling from RF-heated susceptor platter to the satellite is determined by the gas-foil rotation (GFR) gap, thus, the “flying height” of the satellite within the wafer pocket. The flying height h is given by:

$$h = \sqrt[3]{\frac{3\pi \cdot \eta \cdot Q}{\rho}}$$

where Q is the rotation gas flow, η is the dynamic viscosity of the gas and ρ is the density of the satellite material.

Thus, the individual flying height h_x – and consequently the surface temperature – can be modulated by the gas flow Q_x provided to that satellite.

At given process steps, e.g. during the GaN buffer growth, the thermal surface profile ($T_{\text{sat},1..n}$) of all satellites 1..n and the current mean surface temperature (T_{mean}) are measured at one common GFR flow Q_{sat} by in-situ pyrometry. In a second step, by growth recipe control a correction formula is applied to the individual satellite flows $Q_{\text{sat},1..n}$ to achieve the same T_{mean} temperature on all satellites.

Using the AutoSat satellite temperature controller, the temperature variation from satellite to satellite could be reduced from $\Delta T = 4.5$ K down to 0.3 K for a mean temperature around 760°C.

With these promising results a ten run LED marathon with fully loaded susceptor was performed in the AIX G5HT in the 14x4 inch configuration. The wafers were exchanged in fully-automated wafer handler mode and the satellites were baked externally in between runs. Several batches of satellites were in cyclical use to render the experiment equivalent to conditions in a 24/7/365 mass production environment.

All 140 wafers from the marathon were evaluated by photoluminescence (PL) mapping, white light interference (WLI) thickness mapping and XRD.

The total thickness of the LED structures was determined via WLI to 6.21 μm with a standard deviation of $\sigma = 1.05\%$. The distribution was statistical and the results did not show any signs of drift from run to run. XRD measurements exhibited an average well and barrier pair thickness of around 20 nm, also without any signs of drift from run to run.

In this experiment the maximum wavelength spread was 4.9 nm peak to peak at an average of 457.2 nm. The on-wafer standard deviation was around 1.3 nm on average, exhibiting no adverse effects of the GFR tuning on the uniformity of the layers.

Additional results will be discussed and presented, especially the benefits of automated wafer handling and the choice of the in-situ pyrometer for control for the growth of III-nitride materials, nanostructures and devices.

Thermodynamic analysis of InGaN-HVPE growth using III-bromides and III-iodides

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InGaN alloys are attracting much attention as potential materials such as optical devices operated in visible spectrum range. However, it is quite difficult to obtain a homogeneous $\text{In}_x\text{Ga}_{1-x}\text{N}$ with high indium content due to the lattice mismatch between InGaN epitaxial layer and substrate and the compositional unstable nature. Consequently, the longest wavelength of nitride lasers has been limited to about 533 nm. Therefore, high quality thick InGaN layers for use as lattice matched substrates are key materials for the fabrication of optical devices operated at longer wavelengths.

Halide vapor phase epitaxy (HVPE) is one of the most attractive growth processes for fabricating bulk III-nitride crystal because of its high growth rate and low impurity concentration. In our previous thermodynamics analysis, it was shown that InGaN can be grown with high controllability of solid composition when InCl_3 and GaCl_3 are used as group-III precursors [1]. On the other hand, there are other halides of the group-III precursors such as InBr , GaBr , InBr_3 , GaBr_3 , InI , GaI , InI_3 and GaI_3 . It is important to clarify the dependence of group-III precursors on the growth features such as especially the driving force for InGaN growth, controllability of solid composition. In this work, thermodynamic analysis of InGaN alloy growth by reaction between metal-bromides or -iodides, and NH_3 was carried out.

In the calculations, the following nine species were chosen as the necessary gaseous species over InGaN alloy: InX , InX_3 , GaX , GaX_3 , X_2 , HX , NH_3 , H_2 and inert gas (IG) ($\text{X} = \text{Cl}$, Br or I) in bromide and iodide system. The calculation of the equilibrium partial pressures of vapor species was performed using a procedure similar to that described previously [1].

First, changes of the solid composition x in $\text{In}_x\text{Ga}_{1-x}\text{N}$ as a function of the input mole ratio of indium mono-halide precursors ($R_{\text{InX}} = P^\circ_{\text{InX}} / (P^\circ_{\text{InX}} + P^\circ_{\text{GaX}})$ ($\text{X} = \text{Br}$ or I) were investigated. It was found that the solid composition x in $\text{In}_x\text{Ga}_{1-x}\text{N}$ deviates from a linear function and the indium solid composition is almost zero under the wide range of R_{InX} . As for the InGaN-HVPE using tri-halide precursors, we calculated the solid composition as a function of the input mole ratio of indium precursors ($R_{\text{InX}_3} = P^\circ_{\text{InX}_3} / (P^\circ_{\text{InX}_3} + P^\circ_{\text{GaX}_3})$) ($\text{X} = \text{Br}$ or I). As similar to the results of mono-halide precursors, indium solid composition is also almost zero using tri-bromide and tri-iodide, whereas that increases with increasing the input ratio of indium tri-chloride. This is due to the low driving force for InN growth by using In-bromides and In-iodides compared with In-chlorides. These results can be explained by the quite low (3 to 5 orders of magnitude lower) equilibrium constants (K_i) of growth reactions of $\text{InX}(\text{g}) + \text{NH}_3(\text{g}) \rightarrow \text{InN}(\text{s}) + \text{HX}(\text{g}) + \text{H}_2(\text{g})$ ($\text{X} = \text{Cl}$, Br or I) and $\text{InX}_3(\text{g}) + \text{NH}_3(\text{g}) \rightarrow \text{InN}(\text{s}) + 3\text{HX}(\text{g})$ ($\text{X} = \text{Br}$ or I) compared with conventional HVPE system (InCl_3 - GaCl_3 - NH_3 system). From these results, it is clear that the order of driving force is $\text{III} \cdot \text{Cl}_3 \gg \text{III} \cdot \text{I}_3 > \text{III} \cdot \text{Br}_3 > \text{III} \cdot \text{Cl} > \text{III} \cdot \text{I} > \text{III} \cdot \text{Br}$, and the HVPE growth of InGaN using compounds of bromine and iodine is very difficult. In other words, the only way to HVPE growth of InGaN is InCl_3 - GaCl_3 - NH_3 system from the viewpoint of good controllability of solid composition.

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GaN layers growth on pseudo (111)Al substrates by RF-MBE and their chemical lift-off technique

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Fabrication of integrated GaN-based light-emitting devices (LEDs) requires a vertical carrier injection. In the case, GaN growth on metal substrates is one of the solutions. Epitaxial growth of (111)Al on (0001)sapphire was investigated for the application to *pseudo* Al substrates for the III-V growth. The *pseudo* Al substrates have an advantage for the fabrication of GaN-based light-emitting devices with a lift-off process. The chemical lift-off process for the GaN layers grown on *pseudo* Al substrates is also discussed.

At the growth temperatures around 200 °C, the epitaxial growth was achieved. XRD patterns indicated that the (111)Al layers were strained. Thus, we called the (111)Al/(0001)sapphire substrates as pseudo Al substrates. Here, RHEED patterns showed that the azimuth of the grown layers were [1-10]Al//[1-100]sapphire. The results indicate that the epitaxial azimuth relationship between (0001)GaN (or AlN) and (0001)sapphire is dominated by the aluminum absorption on the sapphire. The surface nitridation was also investigated. At the temperature of 350°C, streaky AlN RHEED patterns were observed. XRD patterns indicated that (111)Al layers were still remained after the nitridation. GaN layers were also grown on the pseudo substrates below the melting point of aluminum.

The (0002)GaN, (0002)AlN and (111)Al diffraction peaks were observed in the XRD patterns. We did not observe the formation of stacking mismatch boundaries in the GaN layers. The near-band-edge emission was observed in the photoluminescence spectra at RT. Those results clarified that the pseudo Al substrates grown by MBE have a potential for the application to the light-emitting devices based on III-V nitride materials.

Although the conductive pseudo Al substrates are interesting for the fabrication of integrated GaN-based light-emitting devices, the fabrication of GaN-based LEDs with a chemically lift-off process is a crucial issue for the cost-effective high power LEDs. It was clarified that the chemical lift-off process was easily performed using a diluted HCl solution. This was due to the chemical etch of the pseudo Al substrates.

Strain relaxed growth of n-GaN epilayers

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A significant stress-relaxation was observed in GaN epilayers by integrating a Si-doped sacrificial layer in the un-doped GaN templates grown on sapphire substrates by metal-organic chemical vapor deposition (MOCVD) technique. A hexagonal array of circular SiO₂ dots was patterned on sapphire surface. After patterning, 500nm Si-doped GaN grown in the 1 μ m undoped-GaN template by metal-organic chemical vapor deposition (MOCVD) and SiO₂ masks were removed using buffered oxide etch solution. The n⁺-GaN layer was then electrochemically etched for 2 hr to obtain the desired air-gap structure embedded inside u-GaN layer. After etching, GaN based InGaN/GaN LED structure re-grown sample with embedded air gap inside u-GaN.(Fig 1) Stress-relaxation and its local variations were probed by Raman mapping of high-frequency transverse-optical E_2 (high) phonon mode of GaN. It is well documented that E_2 (high) mode shows a blue shift (red shifts) in presence of a compressive (tensile) strain in GaN. For stress values causing biaxial deformation in the basal plane of GaN within the elastic limit i.e. in the region of Hooke's law (Fig.2). Enhanced In incorporation and improved light emission were observed in InGaN/GaN multi-quantum well (MQW) visible light emitting diode (LED) structures fabricated on strain-relaxed GaN-epilayers with embedded air-gaps. Relevant sources for strain reduction and improved optical emission have been discussed.

Growth and Characterization of the AlGaN epilayers grown on the variation of high temperature AlN buffer layer thickness by HVPE

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Among III-nitride wide-band gap semiconductors, AlN and AlGaN alloy semiconductors have recently attracted much attention due to their applications for high-power and high-frequency electronic devices and optoelectronic devices. For the realization of these devices, the epitaxial growth of high quality AlN and AlGaN with which high aluminum (Al) content is required [1]. For more than few years of AlGaN HVPE development various designs of growth apparatuses and process arrangements were described in numerous scientific publications [2].

The Al_xGa_{1-x}N epilayers were grown by a home-built horizontal HVPE system. The process zone consisted of two parts, which were the source zone and the growth zone. Ammonia (NH₃) gas and a hydrochloric acid (HCl) gas were used as the active gas, and the metallic gallium and aluminum were used as the group III precursors. The substrate used was 2 inch (0001) sapphire. The group III precursors were located in a separated tube at 750 °C for Ga metal and 550 °C for Al metal, respectively, to inhibit the generation of AlCl which degrades the quartz reactor [3]. The GaCl and AlCl₃ gases were generated by reaction with HCl at source zone and transported towards the radiatively heated substrate by N₂ carrier gas. After the nitridation, Al_xGa_{1-x}N epilayers were grown in the growth zone.

First, effect of the growth temperature on the properties of Al_xGa_{1-x}N epilayers grown by HVPE is reported. The Al_xGa_{1-x}N epilayers were grown at different growth temperature from 1050 to 1130 °C at intervals of 20 °C under fixed other growth conditions. Al_xGa_{1-x}N epilayers were grown directly on sapphire without any buffer layers. The Al_xGa_{1-x}N epilayers grown at 1110 °C has the smoothest surface, good transmission among the samples. This result indicates that higher growth temperature is essential to grow a high-quality Al_xGa_{1-x}N epilayer. All the grown Al_xGa_{1-x}N epilayers showed mirror-like surfaces. However, micro-cracks were observed even at the epilayer grown at 1130 °C which showed best crystal quality. So we introduced high temperature AlN buffer layer. The Al_xGa_{1-x}N epilayers were grown with variation of high temperature AlN buffer layer thickness. The buffer growth time was varied from 1 to 3 min at intervals of 1 min. The thickness of AlN buffer layers were 162, 205 and 267 nm, respectively. The crystal quality gets well obviously when the AlN buffer layer thickness change from 162 to 205 nm, but it gets worse when the buffer layer thickness increase to 267 nm. The change of the crystal quality was suggesting that the optimal thickness of the AlN buffer layer on the sapphire substrate was approximately 200 nm. Therefore, the AlN buffer layer thickness is a key factor that can strongly affect the structural properties of the Al_xGa_{1-x}N epilayer. The Al_xGa_{1-x}N epilayer grown on AlN buffer layer of 200nm at 1110 °C showed crack free and good crystal quality.

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Laser patterning – an alternative method to prepare sapphire substrate for GaN growth

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Laser micromachining is a well established technique for fabrication of microstructures [1]. In our work we present use of surface patterning by picosecond laser technique for growth of high quality gallium nitride. Laser ablation is much cheaper and less time consuming technique as photolithography. Quality improvements by use of epitaxial lateral overgrowth (ELO) technique were investigated.

c-plane sapphire surface ablation was accomplished using picosecond Nd:Yag (wavelength 1064 nm) laser, repetition rate – 100 KHz, used wavelength 266 nm (fourth harmonic), pulse duration – 10 ps, sapphire surface ablation power 60-670 mW. Laser ablations made across, in parallel, in 30 degrees and in 120 degrees turn in point to a sapphire plane.

After laser ablation sapphire wafers were cleaned using aqua regia (HNO₃+HCL, in proportion 1:3 respectively) and etched using sulphuric and phosphoric acids solution (H₂SO₄ and H₃PO₄, in proportion 3:1 respectively). SEM images after wet chemical etching shows less surface roughness and it could open sapphire crystalline planes.

Undoped gallium nitride samples were grown by metal-organic chemical vapour deposition (MOCVD). GaN growth conditions: III/IV ratio – 1500, reactor pressure 150 mBar, growth rate ~2,1 μm/h temperature – 1065⁰C. Coalescence layer thickness 1,7 μm, buffer layer thickness 20 nm.

SEM and AFM results shows reduction of dislocation density in gallium nitride layer growth using ELO method on laser patterned (300 mW power) sapphire substrates.

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Stability of GaN(0001) surface coverage at mixed ammonia and hydrogen ambient

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Density functional theory (DFT) is used to simulate the properties of GaN(0001) surface employing a finite slab with additional modifications of the non-real side [1]. Hydrogen adsorption sites and energy, and also the thermodynamic and mechanical stability will be discussed for various surface coverage by hydrogen. It will be shown that adsorption of molecular hydrogen on bare GaN(0001) surface leads to its dissociation and location of H atoms in the sites above the Ga surface atoms with the dissociation energy above 2 eV for hydrogen coverage below 0.75 ML. For 0.75 ML coverage the H₂ adsorption energy is lower than 0.2 eV and process is occurring with energy barrier, which is in agreement with Wampler and Myers data [2]. For higher coverage mechanical stability of hydrogen with respect to molecular desorption is very weak, showing that medium coverage (lower than 0.75 ML) is the most stable. It will be also shown that molecular hydrogen adsorption energy depends in some degree on the electric fields at the surface, i.e. doping in the bulk. Adsorption of ammonia (NH₃) on GaN(0001) surface under various coverage will be also considered, including its transformation to amino radicals (NH₂). The stability of the mixed NH₃-NH₂ coverage is determined both in the mechanical and thermodynamic sense. It will be shown that for high NH₃ fraction, the coverage is mechanically unstable losing hydrogen molecules and also ammonia molecules. The thermodynamic stability will be also considered proving that depending on chemical potentials of hydrogen and nitrogen, the NH₃ to NH₂ ratio is changed. Using kinetic formulation [3], the dependence of the coverage on the partial pressures of ammonia and molecular hydrogen is derived. It is shown that for the conditions corresponding to MOVPE and HVPE growth, the fraction of NH₃ coverage is lower than 40% of lattice sites.

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Hydrogen influence on adsorption of indium and gallium at GaN(0001) surface in MOVPE ambient – ab initio study

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Hydrogen and ammonia adsorption sites and energy, and also the thermodynamic and mechanical stability at GaN(0001) surface under various coverage will be also determined from ab initio DFT calculations. It is shown that ammonia adsorption at bare and hydrogen covered GaN(0001) surface proceeds without any energy barrier. The adsorbed ammonia molecules undergoes transformation to amino radicals (NH₂) by losing hydrogen excess via evaporation to the vapor phase. The energy barrier for the molecular hydrogen desorption process is very low, of order of 0.15 eV, therefore at MOVPE growth conditions, the surface ammonia molecules are transformed to NH₃-NH₂ coverage. It is shown that for high partial pressure of ammonia chemical composition of mixed NH₃-NH₂ coverage strongly depends on the partial pressures of molecular hydrogen. Using kinetic arguments and DFT data, the fraction of NH₃ coverage for the conditions corresponding to MOVPE growth is determined. From ab initio DFT simulations, gallium and indium desorption energy barriers at mixed NH₃-NH₂ coverage are derived. It is shown that indium stability depends more strongly on NH₃-NH₂ ratio, proving that the presence of hydrogen affects adversely indium stability at GaN(0001) surface, decreasing incorporation of indium for higher molecular hydrogen content in the vapor.

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Polarity inversion mechanism at oxidized AlN layers

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Control of polarity for *c*-plane group-III nitrides grown on foreign substrates is inherently important for fabrication of both optical and electronic devices. Several groups have reported on the polarity flip from N-polarity to metal-polarity by inserting oxidized AlN layers.[1,2] However, the mechanism of this phenomenon has been unclear and the degradation in crystalline quality by the insertion of this layer has also been reported.[1] In this study, we have developed a technique that allows us to change the polarity reproducibly and investigated its polarity inversion mechanism.

All the nitride films were grown by pulsed sputtering deposition (PSD). 50 nm-thick AlN epilayers were grown on the N-polar GaN/Sapphire(0001) structure. To oxidize the N-polar AlN surface, the samples were removed from the PSD chamber and then annealed at 500°C in air for 3 h. Structural properties of the oxidized AlN (AlO(N)) layers were investigated by XPS and RHEED.

The film thickness of the AlO(N) layer was determined to be 1.0 nm by the ratio of the XPS Al 2*p* core level peak intensity for the AlO(N) layer to that for the AlN underlayer. RHEED images of this sample with e-beam parallel to [11-20]_{AlN} and [10-10]_{AlN} show bright spotty patterns of AlO(N) superimposed on streaky patterns of the AlN underlayer, indicating that the AlO(N) layers are epitaxially formed on the AlN surface. Careful analysis of their RHEED patterns also implies that the AlO(N) layer possesses the corundum structure and that the epitaxial relationships between them are (0001)_{AlN} || (0001)_{AlO(N)} and [11-20]_{AlN} || [10-10]_{AlO(N)}. We have confirmed that the polarity of AlN reproducibly changes from N-polarity to Al-polarity by the present epitaxial AlO(N) insertion technique with the minimum oxide thickness to avoid the degradation in crystalline quality. This polarity flip can be explained probably by this corundum structure of the AlO(N) epilayer, which does not have the inversion symmetry. Detailed inversion mechanism will be discussed.

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Indium incorporation during growth of InGaN on (0001) surface

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Growth of indium gallium nitride on (0001) surface is a process controlled by surface kinetics of Ga adatoms and occurs in N-rich state. In the supersaturated state kinetics of two consecutive steps of (0001) surface is different. We propose the model that describes the surface of growing InGa crystal at desoriented (0001) surface [1,2]. Ga adatoms are adsorbed at hexagonal lattice sites and diffuse over the surface until they stick to the step. Two different constants describing Ga atoms interaction are assumed. The first one represents two particle Ga-Ga forces, and the second one describes bond of the tetrahedral structure of four Ga atoms. Four-particle interaction is much stronger than the sum of two-particle bonds. Indium atoms are adsorbed at the lattice sites in the same way as gallium atoms. They desorb immediately after adsorption, unless adjacent Ga atoms pin them to the surface. Adsorption and desorption of In are described by two additional parameters. Geometry of In-Ga forces is the same as Ga-Ga ones. Simulations are carried in N-rich state, hence nitrogen atoms saturate all dangling bonds available for them, and they do not influence the growth. Their presence is represented by geometry of bonds between metallic atoms.

We performed extensive kinetic Monte Carlo simulations of the model by using CUDA programming of graphic cards. During crystal growth simulations we investigated influence of gallium and indium flux and temperature on amount of incorporated In atoms. When particles are incorporated very slowly we have parallel steps but very little In atoms built in. High concentration of incorporated indium leads to surface roughening.

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High-quality AlN layer deposited at high substrate temperature with high sputter power by RF reactive sputtering

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Aluminum nitride (AlN), that is one of III-V semiconductors, has useful properties such as wide bandgap (6.2 eV) and high thermal conductivity ($>200 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$). It is demanded as substrate material of ultra-violet LED. In the previous study, the influence of N_2 gas flow ratio and sputter power on crystalline quality of AlN layers sputtered on nitrided c-plane sapphire substrates were investigated at the substrate temperature of 573-823 K [1, 2]. The nitrided sapphire substrate, which was prepared by “sapphire nitridation method”, has high-quality AlN thin single layer with 10 nm thickness on its surface [3]. The purpose of the present study was to optimize the sputter condition with high sputter power of 900 and 1000 W at substrate temperature of higher than 823 K for preparation of high-quality AlN layer.

The AlN layers were deposited by magnetron radio-frequency (RF) reactive sputtering using a pure aluminum target (99.999 mass%) with 900 and 1000 W in N_2 -Ar gas mixture (N_2 gas flow ratio: 40 and 50 vol% N_2). The substrate temperature was varied from 823 to 923 K. The 2θ - ω profile of AlN sputtered layers is observed by X-ray diffraction (XRD) due to phase identification. The full width at half maximum (FWHM) of AlN (0002) and (10-12) XRD rocking curve (XRC) was measured to evaluate c-axis orientation (tilt and twist). The surface of these layers was observed by a laser microscope.

In 2θ - ω profile of AlN layer sputtered in 40 vol% N_2 with 1000 W at 923 K, AlN (0002), (0004) and (10-10) diffraction peaks were observed, which indicates mix-oriented AlN layer is grown in this condition. On the other hand, only AlN (0002) and (0004) diffraction peaks were observed in other sputtering conditions, which indicates that c-axis oriented AlN layers are grown. The position of AlN (0002) diffraction peaks for AlN sputtered layers shifted to lower degree with increasing substrate temperature, which indicates that AlN layers sputtered with higher substrate temperature have higher residual stress. The intensity of AlN (0002) diffraction peak for c-axis oriented AlN layers sputtered at 923 K (in 40 and 50 vol% N_2 with 900 W; in 50 vol% N_2 with 1000 W) was weak and almost same as that of nitrided sapphire substrate. These AlN layers were partially peeled from a substrate. Therefore, these AlN sputtered layer probably have excessive residual stress.

The XRCs for c-axis oriented AlN layers, which were grown without peeling, sputtered at 823 and 873 K were measured. The AlN layer sputtered in 50 vol% N_2 with 900 W at 823 K achieved the best orientation quality, and their AlN (0002) and (10-12) XRC-FWHM are 61 and 864 arcsec, respectively. These values are almost same as the nitrided sapphire substrate (AlN (0002) XRC-FWHM: 40-100 arcsec, AlN (10-10) XRC-FWHM 700-1000 arcsec). Thus, c-axis oriented AlN layers without peeling were successfully grown with 900 W at 823 and 873 K by a RF reactive sputtering method.

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Spatial stress distribution of GaN grown on a serpentine masked structure

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High-quality GaN grown on a serpentine masked structure (SMS) was obtained. Compared to the conventional epitaxial lateral overgrowth (ELOG) mask, the developed serpentine mask has demonstrated advantages for material growth and device fabrication. From the use of the SMS, we can expect that the properties of a GaN film are different in view of local stress distribution compared to that of the GaN film grown on a conventional ELOG mask. In order to investigate spatial variations in stress in the GaN epilayer, we performed micro-Raman (μ -Raman) line-scan measurements. Raman spectra were extracted from the wing, window and meeting front regions. It is noticed that the intensity of wing and window regions is much higher than that of meeting front, indicating a higher GaN crystalline quality at these two regions. The E_2 (high) phonon mode at the wing and window regions of the GaN surface are almost the same, at $\sim 570.5 \text{ cm}^{-1}$, while a lower value $\sim 569.9 \text{ cm}^{-1}$ in the meeting front region. Compared to the bulk value of 567.6 cm^{-1} at room temperature, all the peak values of E_2 (high) phonon modes are shifted upward, indicating that the GaN layer is compressively stressed, as expected in GaN grown on sapphire substrates. The in-plane biaxial stress was calculated to be 0.69 GPa and 0.55 GPa in the wing, window and meeting front regions, respectively. The results implied that the interface force introduced by the mask was too small to vary the strain between the wing and window regions. It quite differs from the stress distribution of GaN using conventional ELOG method in which strain is different for each region. To clarify the mechanism, x-ray diffraction (XRD) (002)-plane ω -scan with the scattering plane perpendicular to the stripe direction was done. There was only one peak which means no tilt occurred in the wing area, representing that the SiN mask didn't lead to the variation of crystal orientation between the window and wing region. It was attributed to the quite lower fill factor (the width ratio of the upper window to the period) 0.1 in this experiment, which resulted in a uniformly strained GaN films. We argue that the stress relaxed in the form of dislocations in the meeting front region; in addition, strain related to dislocations is often tensile, resulting in a decrease in the E_2 (high) phonon mode energy. Therefore, the compressive strain in the meeting front region is smaller than the other two regions. The results are helpful for us to design high performance GaN-based optoelectronic and electronic devices on the SMS.

Peculiarities of fast AlGa_N growth in planetary reactor

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Thick AlGa_N layers with high Al content are of great interest for various device applications. However, strong parasitic reactions between TMAI and NH₃ limits AlGa_N growth rates in conventional multi-wafer MOVPE systems at relatively low level. The problem may be partially solved by using specially-designed reactors [1] or strong reduction of chamber height in CCS reactors [2]. In the presented work we have investigated the possibility of fast AlGa_N growth in the conventional 6*2" AIX2000HT planetary reactor with relatively long residence time by adjusting gas flows for minimization of parasitic reactions effect on growth efficiency.

Recently we have reported AlN growth rates up to 8.6 μm/h in planetary reactor using very low NH₃ flows [3]. However, low gallium incorporation efficiency at these conditions does not allow to grow AlGa_N with Ga content above a few percent which make a gap between conditions for fast AlN growth (low NH₃ flow) and fast Ga_N growth (high NH₃ flow). Investigation of AlGa_N growth rate dependences on NH₃, TMGa and TMAI flows has revealed following:

- At low TMGa flows AlGa_N growth rate monotonically decrease with NH₃ increase. For higher TMGa four regions may be distinguished where growth rate is independent, rise, saturates and falls with NH₃. In the first region growth rate is also nearly independent on TMGa.
- At low NH₃ flow increase of TMAI flow results in increase of Ga_N partial growth rate, while at high NH₃ flow the tendency is opposite.
- The dependencies of AlGa_N growth rate on TMGa flow are linear for all investigated conditions but the slope depends on TMAI and NH₃ flows.
- At low NH₃ flow AlGa_N growth rate is higher than the sum of AlN and Ga_N growth rates at the same conditions while at high NH₃ flow AlGa_N growth rate is lower than the sum of AlN and Ga_N growth rates.

These tendencies result from the interplay of 2 effects:

- Surface chemical process of Ga-containing nitrides decomposition under hydrogen-containing ambient strongly depended on NH₃ and H₂ partial pressure and suppressed by TMAI;
- Gas phase parasitic reaction between TMAI and NH₃ resulting in Al-losses due to AlN nanoparticles formation and well-known for AlN growth. In the case of fast AlGa_N growth under high TMAI and NH₃ flows this process results also in Ga-losses due to mixed AlGa_N particles formation [4].

The mechanisms of these effects influence on AlGa_N growth will be discussed.

Adjustment of reactor conditions let us to reach growth rates of 2-3 μm/h AlGa_N layers in the whole composition range and 5-6.5 μm/h in the range of Al content 25-50%.

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Enhancement of In-incorporation into InGaN by nitridation of sapphire substrate in MOVPE

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The growth of In-rich InGaN without the phase separation is required for fabricating brilliant LEDs and LDs with a low-threshold current in the green region. InGaN is a mixed crystal of GaN and InN. For growing In-rich InGaN, it is necessary to efficiently incorporate InN with the highest equilibrium-vapor-pressure of nitrogen among three binary alloys of nitride semiconductors. In other words, the highly efficient incorporation of nitrogen is indispensable to grow In-rich InGaN. Authors have proposed that the N-polar growth is suitable for incorporating nitrogen atoms into a film because one nitrogen atom is captured with three gallium atoms in the N-polar growth. On the other hand, in the growth with the polarity of the group-III, one nitrogen atom is captured with only one group-III atom. Authors have also shown the epitaxially growth of N-polar GaN with the same characteristics such as dislocation density and p-type conduction by Mg-doping as those of Ga-polar GaN. In this paper, the effect of N-polar growth on In-incorporation in the InGaN growth is experimentally confirmed.

For the N-polar growth on sapphire substrates, the process of “nitridation” is brought just before the growth. This nitridation forms an AlN thin layer on the top surface of a sapphire substrate. The nitridation of a c-plane sapphire substrate was carried out at 1040°C for 3 min in the atmosphere of ammonia. Successively, InGaN films were directly grown at 800°C. In comparison, InGaN was also grown on sapphire substrates without nitridation.

To investigate the effect of the nitridation on the indium incorporation, the accurate composition of InGaN must be accurately evaluated. The compositions of InGaN were determined by using reciprocal space mapping (RSM) of X-ray diffraction from (10 $\bar{1}$ 5) asymmetric reflection. The indium content of InGaN grown with and without nitridation was estimated as 13.8% and 7.36%, respectively. This result shows that the nitridation of sapphire has strongly enhanced the incorporation of indium into an InGaN film.

In conclusion, the nitrided sapphire substrate was meaningfully advantageous to the growth of In-rich InGaN. This technique will also open the window for high-quality InN expected as an immerging material for high-speed transistors and temperature-stable LDs in optical communications systems.

High quality GaN layer grown on sapphire substrate using AlN/AlGaIn buffer for UV applications

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GaN layer grown on sapphire substrate with wide band gap AlN/AlGaIn buffer is suitable for application in optoelectronic devices, working in the UV region, because AlN is transparent in that range.

In this work we have investigated the growing of GaN on c-plane Al₂O₃ substrates by low pressure metal organic chemical vapor deposition in planetary horizontal reactor AIX2400G3HT. TMAI and TMGa were used as group-III sources and NH₃ as a group-V source. In all of the steps, the carrier gas was H₂. A growth temperature of 1200°C and chamber pressure of 100-400 mbar were used. The V/III ratio was changed in a range from 50 to 1000.

The structural quality of the epilayers was characterized by X-ray rocking curve measured in reflection (0002) by means of double-crystal diffractometer «Vektor». The concentration of carrier density was measured by Hall method. «In situ» analysis was carried out by means of «LayTec EpiCurveTT».

The influence of lattice mismatch between AlN and Al₂O₃ on quality of AlN was reduced by optimization of growth parameters, such as flow rates of TMAI, NH₃, and V/III ratio. Parasitic reactions between NH₃ and TMAI in a gas phase were minimized by means of increasing the total flow through the reactor. We observed that the quality of AlGaIn/GaN growing on AlN buffer strongly depends on thickness of the buffer. So, the thickness of AlN buffer layer was optimized to 300 nm. Then the n-type Si-doped AlGaIn layer (as n-wide band contact) was grown on this AlN buffer. After that the i-GaN layer and Mg-doped p-GaN layer (as p-contact) were grown. The thickness of n-AlGaIn layer was 800 nm. The thickness of the i-GaN and p-GaN layer was 200 nm and 150 nm respectively. The result of the measurements is shown in the Table 1.

Such kind of buffer layer is suitable for making UV p-i-n photodetector.

Table 1. Result of the measurements

№ Sample	X-ray (0002) FWHM, arcsec	Hall measurements		
		Resistivity, Ohm·cm	Mobility, cm ² /V·s	Concentration, cm ⁻³
AlN	250	-	-	-
n-AlGaIn	450	0,027	70	3·10 ¹⁸
i-GaN	460	-	-	-
p-GaN	460	1,4	5	8·10 ¹⁷

Effect of Dislocation Blocking on HVPE-grown AlN Using the Grooved Seed in Triangle-shaped Stripe

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AlN is useful as a substrate of the AlGaIn-related deep-ultraviolet light emitting device with high efficiency. The growth method to control lateral growth rate using the grooved substrate is promising, because the selective growth of AlN and AlGaIn cannot use SiO₂ as a mask. In this study, the controlled propagation and blocking of the threading dislocations for the purpose of reducing dislocation density was realized by a thick AlN film grown by the HVPE using the AlN/sapphire substrate that has the grooved seed AlN in the triangle-shaped stripe.

The SiO₂ stripe pattern $\langle 1-100 \rangle$ parallel to AlN direction was formed by the photolithography technique, then etched SiO₂ mask with its cross-sections having triangle shape. After that the triangle-striped AlN on sapphire substrate was formed using the SiO₂ mask by dry etching with inductively coupled plasma. The widths of triangle-striped AlN and flat AlN were 3 μm and 7 μm, respectively. The HVPE growth was implemented on the substrate. The growth condition is under the pressure of 5 kPa with temperature of 1450-1500 °C.

The crack-free of 28 μm was obtained by growth for 180 min. The formation of the crack was inhibited because the stress was relaxed by the void formed by lateral growth. In addition, atomic-steps can be clearly observed, suggesting that dislocation density is largely reduced. The RMS value showing surface flatness is 0.121 nm, showing its extremely superior flatness. FWHM value by its X-ray rocking curve was 176 arcsec as for the diffraction (0002), and was 241 arcsec as for the diffraction (10-10), thus turned out the fact of improved crystal characteristics of the twisted ingredient particularly in comparison with pre-processing. Additionally, the evaluation of the threading dislocation density in AlN epitaxial layer was performed by the etch-pit method. As a result of having performed wet etching by the mixed melt of KOH and NaOH at 350-400 °C for 60 seconds. The formed etch pits could class types of dislocation by those size. As for the *a*-type dislocation of AlN which grew up on a flat AlN/sapphire substrate, its density was $1.9 \times 10^9 \text{ cm}^{-2}$, as for the *a+c*-type dislocation, its density was $2.3 \times 10^8 \text{ cm}^{-2}$. For the AlN film grown on the triangle striped AlN sapphire substrate, the *a*-type dislocation density was $7.3 \times 10^8 \text{ cm}^{-2}$, the *a+c*-type dislocation density was $4.2 \times 10^6 \text{ cm}^{-2}$, thus the reduction of threading dislocations to the surface was done successfully to compare with AlN grown on a flat AlN/sapphire substrate. The transmission electron microscope (TEM) observation revealed that the dislocation to propagate from the groove terminated in the lower void and the dislocation was bent by the lateral direction growth from the triangle-shaped side.

We have found new mechanism of reducing threading dislocations in AlN; the dislocations c-plane, which were originated from interface of sapphire and AlN can be blocked at inclined crystal plane, and were bend along the stripe direction of $\langle 1-100 \rangle$, so that the reduction of threading dislocations at the surface was realized.

HVPE growth of a-plane (Al,Ga)N layers on patterned c-plane sapphire

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Fabrication of high-efficiency (Al,Ga)N-based devices operating in the UV-range requires formation of UV-transparent Al-rich (Al,Ga)N buffer layers with low dislocation density. One of the promising methods for the growth of thick bulk-like III-nitride layers for subsequent device epitaxy is hydride vapour phase epitaxy (HVPE). The HVPE growth of thick c-plane ternary (Al,Ga)N layers has been already demonstrated. However, the material quality was not sufficient so far. The necessary defect reduction can be achieved by epitaxial lateral overgrowth (ELOG), which is also known to allow for growth of crack-free layers with higher thicknesses and hence further defect reduction by mutual annihilation. Thus, in our experiment we combined ELOG with HVPE to grow 15 μm thick Al-rich (Al,Ga)N layers.

In our experiment 600 nm thick c-plane oriented AlN layers were grown first by MOVPE on sapphire substrates. The samples were subsequently patterned using lithography and plasma etching. In particular, 1.5 μm deep trenches with a trench width of 5 μm and a ridge width of 3 μm were prepared. The ridges were oriented along the $[1\bar{1}00]\text{Al}_2\text{O}_3$ direction, which results in a-plane (i.e. $(11\bar{2}0)\text{Al}_2\text{O}_3$) trench sidewalls. This is different to most experiments published up to now discussing growth of c-plane oriented (Al,Ga)N layers on patterned sapphire with $[11\bar{2}0]$ ridge geometry. It was shown that during the HVPE growth on the $[11\bar{2}0]$ -patterned sapphire formation of polycrystalline (Al,Ga)N on the m-plane sidewalls of the trenches can occur. Obviously, the preferential growth of polycrystalline regions strongly degrades the quality of the layers. We will show that also for the perpendicular ridge direction, along $[1\bar{1}00]\text{Al}_2\text{O}_3$, growth is dominated by the nucleation at sidewalls of the trenches and that growth of uniformly oriented a-plane (Al,Ga)N layers becomes possible.

Structural analysis by transmission electron microscopy (TEM) showed that c-plane oriented (Al,Ga)N is formed on the ridges with a patterned c-plane AlN seed layer and deep in the trenches on the $(0001)\text{Al}_2\text{O}_3$ surface. In addition, the nucleation directly on the trench sidewalls, results in formation of (Al,Ga)N with $(0001)[11\bar{2}0](\text{Al,Ga})\text{N} \parallel (11\bar{2}0)[0001]\text{Al}_2\text{O}_3$, i.e. a-plane (Al,Ga)N orientation with respect to the horizontal growth front. The growth rate of a-plane (Al,Ga)N exceeds that one of c-plane regions, which leads to the complete overgrowth of c-plane (Al,Ga)N by the a-plane oriented material. As result, a thick continuous single crystalline a-plane (Al,Ga)N layer can be formed. Typical for the ELOG grown layers, there is a non-uniform defect distribution on the layer surface with regions of a high dislocation density appearing at the positions where two side fronts of the growing material coalesce. Furthermore, basal plane stacking faults were observed in the layers.

ω -rocking curves recorded by X-ray diffraction at the symmetric 110 reflection reveal a relatively small range of FWHMs ranging between 350'' and 420'' for a sample rotation around the surface normal. This indicates only a low lateral anisotropy of the a-plane grown material.

Z-contrast STEM and EDXS analyses reveal self-ordering effects, which appear at the beginning of the layers growth. These effects occur due to different diffusion and incorporation efficiency on different nonplanar facets present during the first growth stages, but do not influence the composition of the coalesced planar layers.

Substrate bias effect in AlN buffer layers for InN films deposition by RF reactive sputtering

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Indium Nitride (InN) has received considerable interest due to its potential applications in optoelectronic devices such as high-efficiency solar cells or opto-chemical sensors. Due to the InN low dissociation temperature (~500°C), radio frequency (RF) reactive sputtering is an appropriate growth technique enabling its deposition at low temperature and on flexible substrates.

In this work, we investigate the influence of Aluminum Nitride (AlN) buffer layers (~30 nm thick) deposited under unbiased and biased substrate conditions (us-AlN and bs-AlN buffer, respectively) on the structural and optical properties of the subsequent InN layers. The InN samples under study have a nominal thickness of ~600 nm and are grown on c-axis oriented sapphire substrate by RF reactive sputtering. The use of us-AlN and bs-AlN buffer layers is compared to a low-growth rate InN buffer. The structural quality of the InN layers is assessed by high-resolution x-ray diffraction (HRXRD) and the surface morphology is investigated through atomic force microscopy (AFM). Linear optical transmission measurements are carried out in order to evaluate the optical properties of the samples.

X-ray diffraction measurements reveal a c-axis preferred growth orientation for the InN layers, regardless the buffer layer. The morphology of InN evolves from columnar-like in the case of InN buffer layer to compact film when using the AlN buffer, which indicates a change in the nucleation process. Compared to InN grown using InN buffer, the InN layers on us-AlN show an increase of the grain size from ~ 35 nm to ~ 47 nm and a decrease of the full-width at half-maximum (FWHM) of the rocking curve of the (0002) x-ray reflection from 2.4° to 1.5°. The InN layers deposited on both buffer layers show high values of rms surface roughness, although it is reduced from 71 nm for InN buffer to 50 nm for layers deposited on us-AlN buffer. Structural properties are further improved when using the bs-AlN buffer. Buffer deposition under -15 V substrate bias results in InN layers with a FWHM of the (0002) rocking curve as low as 1.2°, a grain size of ~ 43 nm and an rms surface roughness of 33 nm. This structural characterization indicates that the mosaicity of the InN can be effectively controlled by the AlN buffer. It must be pointed out that preliminary results obtained with a double AlN buffer layer (biased followed by unbiased) leads to InN films with a decreased rms surface roughness of 6 nm.

Concerning the optical properties, InN layers grown using us-AlN or bs-AlN buffer layers present an optical absorption edge at room temperature at about 1.71 eV, red shifted in comparison to the 1.74 eV estimated for the samples with low-growth-rate InN buffer.

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Study of AlN layers grown on sapphire by RF reactive sputtering by a two-step deposition method

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Features like wide-band-gap energy (6.2 eV), high acoustic velocity and strong piezoelectricity make Aluminum Nitride (AlN) an attractive semiconductor for application in UV optoelectronics, surface acoustic wave devices (SAW) and microelectromechanical systems (MEMS). In most of these fields, AlN layers with high crystal quality and smooth surface morphology are required. Radio frequency (RF) reactive sputtering is a low-cost solution which allows the deposition in a wide range of temperatures and substrates. The aim of this work is to investigate the influence of deposition parameters such as deposition pressure (P_c), RF power (P_{RF}), and substrate temperature (T_{sub}) on the crystal quality and surface morphology of AlN films (~120 nm thick) deposited on *c*-sapphire substrates by RF sputtering.

The AlN deposition parameters under study were optimized under unbiased substrate conditions, obtaining layers which show rms surface roughness of (0.6±0.2) nm in a 1×1μm² area, average grain size around 40 nm, and full-width at half-maximum (FWHM) of the rocking curve for the (0002) AlN reflection of 1.9°. The adequate tuning of the substrate bias during deposition is a critical parameter to improve the crystalline quality. The effect of bias was studied in layers deposited under optimized conditions, namely $P_c = 3.5$ mTorr, $P_{RF} = 150$ W, and $T_{sub} = 450^\circ\text{C}$, in pure nitrogen atmosphere. For the considered range of substrate bias (+5 V to -25 V), the best results were obtained under -15 V bias, leading to AlN layers with larger grain size (~55 nm) and reduced FWHM of the rocking curve for the (0002) AlN reflection to 1.35°. In the voltage range under study, we do not observe any variation of the deposition rate with bias, which implies that the observed improvement in structural quality of the layers can be attributed to an increase of the kinetic energy of the impinging ions. At the same time, AlN samples deposited under biased substrate conditions show an rms surface roughness in the same range as the unbiased layers. However, $\theta/2\theta$ scan XRD measurements reveal lattice disorder at the AlN/sapphire interface, which produces a broad reflection at ~35°. Real-time measurements show a transitory elapsed time where the arrival of positively charged ions compensates the externally induced negative bias at the substrate. Thus, the lattice disorder observed is assigned to surface damage by the highly accelerated ions during this transient time. Therefore, in order to prevent the lattice disorder observed in biased layers, a two-step deposition method is developed, which consists of the deposition of a first AlN thin (~30 nm) buffer layer under unbiased substrate conditions, which reduces the length and magnitude of the observed current transient, followed by a biased AlN (~120 nm thick) layer at -15 V. The structural analysis of the layers grown by this method show that the lattice disorder observed in biased layers is successfully eliminated.

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Temperature dependence of a-plane GaN low angle incidence microchannel epitaxy grown by ammonia-based metal-organic molecular beam epitaxy

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Several advantages of a-plane GaN based devices have been reported, such as eliminations of quantum-confined Stark effect (QCSE) and c-plane stacking faults, which enhances an internal quantum efficiency of lasers compared to c-plane GaN based devices. Nevertheless, a-plane GaN layers grown on r-plane sapphire still involve high density of TDs, which is as large as 10^{10} cm⁻². Therefore, several articles have reported reduction of TDs using epitaxial lateral growth or patterned substrates. Unfortunately, wide lateral growth can only be achieved only after growing thick epitaxial layers, because conventional MOCVD has a very weak growth rate dependence on the crystal orientation. Hence, we have employed NH₃-based metal-organic molecular beam epitaxy (MOMBE) to suppress the vertical growth and to obtain a wide lateral growth. The lateral growth is performed using low angle incidence microchannel epitaxy (LAIMCE), which is a special technique to achieve lateral growth by MBE by supplying molecular beams in a very low incidence angle to the substrate [1]. In this paper, the growth temperature dependence on the lateral growth is especially studied to optimize GaN LAIMCE on an a-plane GaN template.

All samples were grown on GaN templates on sapphires by using NH₃-based MOMBE. Openings with a width of 5 μm and a period of 30 μm were cut in the SiO₂ mask on the template in the [1-100] direction using a conventional photolithography. TMG and NH₃ were used as the precursors for Ga and N, respectively. TMG was supplied through a nozzle with a low incidence angle of 5° relative to the substrate plane while NH₃ was supplied with an angle of 45°. While [NH₃]/[TMG] ratio and time were set at 15 and 6 h, respectively, growth temperature was varied from 780 °C to 860 °C.

In all temperature range, thin lateral growth with flat surfaces were obtained. Though a very few GaN polycrystals were observed on the mask at 780 °C, they completely vanished at 860 °C. A white stripe on the front side of the epitaxial layer grown at 780 and 820°C indicates that Ga adatoms migrated from the side to the top by the formation of {11-22}GaN facets on the side. On the contrary, the wide lateral growth with very flat top of the sample grown at 860°C suggests that the reverse direction of inter-surface diffusion had occurred and it enhanced the lateral growth in the direction of the precursors supply.

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Initial growth stages of AlN/c-Al₂O₃ by molecular beam epitaxy

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High threading dislocation (TD) density (up to $\sim 10^{10} \text{cm}^{-2}$) is one of the most serious problem in Al_xGa_{1-x}N layers with $x > 0.3$, grown on c-sapphire. TD's generated during the initial growth stages of AlN buffer layers on sapphire substrates can reach active regions of devices causing their degradation. This paper reports on the role of a grain structure of the AlN nucleation layers (NL) in both reducing the TD's density in Al-rich AlGa_n heterostructures and generation of an additional elastic stress.

Experimental samples with three types of the AlN/Al₂O₃ NL's were grown by PA MBE. The first one was grown at a low substrate temperature $T_s = 550^\circ\text{C}$ (LT mode), while other two at high temperatures (HT) $T_s = 750\text{-}800^\circ\text{C}$ by using either a continuous mode (HT-C) or a migration enhanced epitaxy (HT-MEE) with alternating supplying of the Al and N fluxes for 30 sec each. The N-rich conditions were used for growth of both the LT and HT-C NL's having a thickness of 130 nm. The thickness of HT-MEE NL was 50 nm. All the structures were terminated with (1-2)- μm -thick 2D AlN buffer layers grown in a pulse Al supplying mode under the Al-rich conditions [1]. Finally, identical Al_{0.5}Ga_{0.5}N(3nm)/Al_{0.6}Ga_{0.4}N SQW structures were grown atop for the comparison. RHEED, laser reflectometry were used to characterize the structures. The samples were studied by using atomic force and transmission electron microscopies, cathodoluminescence (CL), and X-ray diffraction analysis of both the symmetric AlN(0002) and screw symmetric AlN(10 $\bar{1}$ 5) reflexes.

The widest FWHM value for AlN(0002) of 1944 arcsec was measured for the LT-NL, while the HT-C and MEE-HT NLs exhibited much less values of 774 and 469 arcsec, respectively. The AlN(10 $\bar{1}$ 5) reflexes showed the corresponding FWHM values of 2910, 1194 and 1025 arcsec. The densities of the screw(edge) TD's were evaluated to be as high as $8.4 \cdot 10^9 (4.2 \cdot 10^{10}) \text{cm}^{-2}$ in the LT-sample, while for HT-C and HT-MEE samples the TD densities were reduced noticeably to $1.3 \cdot 10^9 (7 \cdot 10^9) \text{cm}^{-2}$ and $4.7 \cdot 10^8 (6.2 \cdot 10^9) \text{cm}^{-2}$, respectively. These results can be explained by the different grain sizes in the NLs which were governed mainly by their growth temperatures, with the lowest grain diameters of ~ 30 nm being found in the LT-NL. In contrast, the HT growth (especially in MEE mode) led to extending the grains sizes found by AFM and RHEED, which resulted in decrease of TD's densities. The strong effect of TDs on the radiative efficiency was confirmed by the highest intensity of the CL peak at ~ 280 nm in the SQW structure grown atop of the HT-MEE NL.

The evolution of lateral stress in the thick 2D HT-AlN buffer layers with different NLs has been studied using RHEED. Only the layer with LT-NL demonstrated sharp increase/decrease of an a-lattice constant by $\Delta a/a \sim 3\%$ after closing/opening the Al-source. These effect seems to be related to the incorporation of Al adatoms into the grain boundaries during growth, with the LT-NL sample having the largest boundary length. This led to generation of elastic compressive stresses in the c-plane, which were released during the growth interruptions similar to the case reported in Ref. [2].

Thus, the AlGa_n-based SQW structure with the lowest TD's densities and highest RT CL peak intensity at 280 nm was grown by using HT-MEE NL possessing the relatively wide and flat grains. The significant role of the grain boundaries in the generation of strong additional compressive stresses has been demonstrated as well.

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Highly n-type conductive high Al-content Al_xGa_{1-x}N (x>0.7) grown by hot-wall MOCVD

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While n-type GaN is easily obtained, growth of conductive AlGa_xN alloys with high Al-content is more difficult. The difficulties have been argued to be due to increased activation energy of the Si donor and/or possible transition from shallow donor to deep DX centers of silicon and oxygen, as well as carrier compensation by cation vacancy-related acceptors which are suggested to have low formation energies. Moreover, the carrier mobility has been reported to decrease significantly with increasing Al content. While great progress has been reported for materials with Al content below 70%, there are few investigations on conductive AlGa_xN with Al content exceeding 80% using MOCVD [1,2].

In this paper, growth, structural, optical and electrical properties of highly n-type conductive silicon doped Al_xGa_{1-x}N (x>0.7) layers grown by hot-wall MOCVD are discussed. An electron concentration of $\sim 2 \times 10^{18} \text{ cm}^{-3}$ and with an electron mobility of $82 \text{ cm}^2/\text{Vs}$ was achieved for Al_xGa_{1-x}N layers with x~0.8 at a moderate Si doping level of $3 \times 10^{18} \text{ cm}^{-3}$. This corresponds to a resistivity of $0.035 \text{ } \Omega\text{cm}$ at room temperature which is consistent to state-of-the-art data [1] but at one order of magnitude lower silicon doping level. At these moderate doping levels thick Al_{0.8}Ga_{0.2}N can be grown with high structural quality and high electron mobility.

The conductive Al_{0.8}Ga_{0.2}N layers are characterized by a smooth step like surface with a RMS roughness of 0.3 nm ($2 \times 2 \text{ } \mu\text{m}^2$) and room temperature cathodoluminescence shows intense near-band-gap emission at $\sim 231 \text{ nm}$. Temperature-dependent Hall measurements indicate that silicon is a relatively shallow donor and conduction is observed at low temperatures ($\sim 100 \text{ K}$). Furthermore, electron paramagnetic resonance spectrum is observed in darkness at low temperatures (4-100 K), strongly supporting that silicon is a shallow donor in AlGa_xN alloys with an Al-content of 80%. The carrier concentration determined from transport measurements agrees with the Si atom concentration in the layers as measured by SIMS indicating low compensation in the material for the particular growth conditions. The electrical properties of Si-doped Al_xGa_{1-x}N layers will be discussed relative growth parameters, including Si and impurity incorporation.

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Plasma-assisted electroepitaxy of GaN layers

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Solution growth methods are normally used in the commercial production of standard III-V crystals with low dislocation density. It is well established that by using solution growth techniques, the crystallisation takes place very close to equilibrium. However, the solubility of N in liquid Ga is very low and therefore it is difficult to obtain reasonable growth rates of GaN from liquid Ga.

In conventional plasma-assisted molecular beam epitaxy (PA-MBE) it is now well established that the highest quality GaN layers can be grown under Ga-rich conditions. Whilst the solubility of molecular nitrogen in Ga under normal growth conditions is very low, because the process is dissociative with a high energy barrier; the solubility of atomic nitrogen in Ga is expected to be relatively high [1]. Therefore atomic nitrogen from a plasma source can be used to produce high concentrations of N in a Ga-based melt. Unfortunately, this high concentration of N only exists close to the surface of the metallic Ga and normally results in spontaneous crystallization of polycrystalline GaN on the surface of metallic gallium. In order to achieve an efficient epitaxial growth process one needs to develop a technique to transport the N species through the gallium melt to the growth surface and at the same time to minimize spontaneous nucleation.

Liquid phase electroepitaxy (LPEE) is a crystal growth method, in which the layer growth is initiated and sustained by passing a direct (DC) electric current through the solution-substrate interface while the temperature of the overall system is maintained constant [see review 2].

We have studied a novel approach for the growth of GaN layers, namely plasma-assisted electroepitaxy (PAEE). In this method, we have combined the advantages of the plasma process for producing high concentrations of active N species in the Ga melt with the advantages of electroepitaxy in transferring the N species from the Ga surface to the growth interface, without spontaneous crystallisation on the surface or within the solution. We have designed and built a new vacuum growth chamber which allowed us to combine the plasma-assisted molecular beam epitaxy process with a liquid phase electroepitaxy. We have demonstrated that it is possible to grow continuous GaN layers by PAEE from liquid Ga melt at growth temperatures as low as $\sim 650^{\circ}\text{C}$, with low nitrogen overpressures of $\sim 3 \cdot 10^{-5}$ Torr.

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Characterization of ultrathin InN films grown on YSZ substrates

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InN is one of the most promising materials for high-speed electron devices because of its large electron mobility and high saturation drift velocity. Ultrathin InN films grown on insulating substrates are especially attractive because they are possibly used for fabrication of field-effect transistors. For this purpose, a process technique that allows us to obtain high-quality ultrathin InN films is necessary. However, growth process of ultrathin InN films still suffers from formation of a high density of dislocations and point defects due to the lack of suitable substrate materials. It is known that {111} planes of yttria-stabilized zirconia (YSZ) give a small lattice mismatch of 2.7 % with *c*-plane InN. Therefore, we can expect growth of high-quality ultrathin InN films on YSZ (111) substrates. In this study, we have grown ultrathin In- and N-polar InN films on YSZ (111) and investigated dependence of structural properties and growth mode on the film polarity.

Two types of substrates with different surface atomic compositions were prepared by thermal annealing at 1250 °C. During the annealing, the surfaces of the substrates were capped with a YSZ wafer (substrate A) or a sapphire wafer (substrate B). With atomic force microscopy (AFM), we confirmed that both substrates possess clear stepped-and-terraced surfaces. We grew InN films on the atomically-flat substrates by pulsed sputtering deposition (PSD). Comparing scanning electron microscopic images of the InN surfaces before and after KOH etching, we found that In-polar InN grows on the substrate A whereas N-polar InN grows on the substrate B. Bases on the data of x-ray photoelectron spectroscopy (XPS), we concluded that difference in the amount of the segregated Y atoms at the surfaces is responsible for this phenomenon. These results indicate that polarity of epitaxial InN layers can be controlled by the choice of capping materials during annealing of YSZ substrates.

We grew In- and N-polar InN films with thicknesses of 1 nm - 20 nm on YSZ (111). After removal of excess In metals on the surfaces by HCl solutions, we investigated their surface morphology with AFM. The root mean square of the surface roughness for a 6 nm-thick In-polar InN film grown under a nitrogen-rich condition at 400 °C was 0.2 nm. The film exhibited a clear stepped-and-terraced structure on the surface, which indicates that In-polar InN grows in a two-dimensional mode from the initial stage on YSZ at 400 °C. To evaluate the residual strain of this film, grazing incidence x-ray diffraction (GIXD) measurements were performed. The GIXD curves have revealed that the *a*-axis lattice constant of the film was 0.3548 nm, which suggests that the InN film has tensile strain of 0.3 % compared with the fully-relaxed InN (0.3536 nm). These results indicate that the high quality ultra-thin InN films can be grown on insulating YSZ substrates with the present technique.

Pyrolysis of dimethylhydrazine for the MOVPE growth of GaN and InN monitored by in-situ quadrupole mass spectroscopy

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The use of hydrazine derivatives, instead of ammonia, as the nitrogen precursor in the MOVPE growth is advantageous due to their relatively low pyrolysis temperatures [1,2]. Among them dimethylhydrazine (DMHy) has been attempted successfully in the growth of cubic-III-nitrides and III-V-N type dilute alloys [3,4], where the lower growth temperature is essential to ensure the growth of these metastable films. In our preceding study, InN films have also been grown using DMHy in a specially designed reactor (separate-supply method) to suppress the gas-phase parasitic reactions between DMHy and trimethylindium [5]. The grown film surface, however, was still covered with indium droplets which prevented the growth of thicker films. Although a thermodynamic study predicts DMHy as a potential nitrogen precursor for the InN growth without droplets over a wide range of V/III ratios [2], our results suggest that the growth mechanism is somewhat more complex than the simple model, and details on the gas-phase reactions during the growth are essential in order to grow InN films at higher quality. In this study, the pyrolysis of DMHy in the MOVPE reactor was monitored by an in-situ quadrupole mass spectrometer (QMS) to gain some insight into the gas-phase reactions.

A quartz capillary is inserted into a horizontal LP-MOVPE reactor for the gas sampling, and the sampling position (i.e., the height from the susceptor surface to the capillary orifice) is varied. Another capillary is already introduced for the supply of DMHy in the separate-supply method [5]. Hydrogen and nitrogen are used as the carrier gases.

When the QMS signal intensities of DMHy and the pyrolysis species in N₂ (1.5 slm, 60 Torr) are monitored at the sampling point of 1 mm high from the susceptor surface with varied susceptor temperature (RT-1000°C), DMHy begins to decompose around 400°C and some other species such as H₂, C, CH₄, NH₃...increase their intensities with rising temperatures. These species are believed to be the major end-products of the DMHy pyrolysis. The decomposition rate of DMHy is calculated for temperatures up to 1000°C assuming no thermal decomposition of DMHy at RT. The decomposition rates near the susceptor surface in H₂ and N₂ ambients show that the temperature for the 50% decomposition is 470~600°C, which is increasing with lowering the reactor pressure irrespective of the carrier gases. These values are higher than that (~ 420°C) reported by Lee et al. [6], who performed pyrolysis experiments in an isothermal flow tube reactor at atmospheric pressure in H₂, D₂ and He ambients. These results imply a strong dependence of the pyrolysis rate on ambient pressure, namely, higher pressures induce more efficient decomposition of DMHy. DMHy decomposes less at higher sampling points than in the proximity of the susceptor surface and this tendency is stronger in N₂ than in H₂. This difference is interpreted in terms of the greater thermal conductivity and specific heat of H₂ than N₂.

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Epitaxial growth of semipolar InAlN with high In concentrations on yttria-stabilized zirconia substrates

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The use of InAlN in making nitride heterojunction is quite advantageous because we can increase the band discontinuity while keeping the lattice mismatch small. Semipolar InAlN with high In concentrations is especially attractive for optical applications in the infrared region because it suppresses detrimental effects of the polarization-induced electric fields. However, due to lack of lattice matched substrates, there have been no reports on growth of high-quality semipolar InAlN films so far. Recently, we have found that high-quality InN grows on nearly lattice matched YSZ substrates by the use of pulsed excitation deposition techniques and that its growth orientation can be chosen arbitrarily by controlling plane indices of YSZ. [1, 2] In this presentation, we demonstrate the epitaxial growth of semipolar InAlN with high In concentrations on YSZ substrates by using a pulsed sputtering low temperature deposition technique.

All the InAlN films were grown on YSZ(113) substrates with a pulsed sputtering deposition (PSD) apparatus at growth temperatures ranging from 250 to 500 °C.[3]

We obtained epitaxial InN(1-103) films on YSZ(113) at 350 °C. The epitaxial relationship between InN and YSZ was InN{0001}/YSZ(111) and InN[11-20]/YSZ[-110], which is consistent with the previous report by PLD. [1] However, we have found that In_{0.9}Al_{0.1}N(0001) grows on YSZ(113) at 350 °C, instead of semipolar (1-103) InAlN. Hence, we tried to grow semipolar InAlN on YSZ(113) substrates by inserting a 300-nm-thick InN(1-103) buffer layer and have found that InAlN(1-103) indeed grows. The In content calculated under an assuming of complete lattice relaxation was 86%, which is consistent with data of RT-PL measurements. Although the lattice mismatch between In_{0.86}Al_{0.14}N and YSZ is as large as 4.5%, the insertion of InN between them divides this lattice mismatch into 1.8% and 2.7% for In_{0.86}Al_{0.14}N/InN and for InN/YSZ heterointerfaces, respectively. These facts indicate that reduction in lattice mismatch is inherently important especially for successful growth of semipolar InAlN.

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Growth of GaN layers with low dislocation density using specialized MBE system

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One of the main problems in manufacturing of GaN-based devices up to date is the lack of low cost, lattice matched substrates to III-nitrides. Heteroepitaxy of nitrides on mismatched substrates in spite of using of special procedures at initial growth stage usually gives high dislocation density (up to 10^8 - 10^{10} cm⁻²) affecting on the device quality and reliability. Moreover, typical growth temperatures in MBE are much lower as compared with MOCVD. It leads to insufficient surface mobility of atoms and worse coalescence of nucleation blocks on initial growth stage which, in turn, results in increased active layer dislocation density. Typical values of room temperature electron mobility in GaN grown on sapphire using different buffer layers (usually- thin GaN, AlGa_{0.3}N or AlN layers) are in the range 250-350 cm²/V's for MBE and 500-700 cm²/V's for MOCVD. High value of electron mobility (560 cm²/V's) in GaN grown by MBE on sapphire has been achieved using special buffer layer AlN grown by magnetron sputter epitaxy [1]. One of the best values of electron mobility in GaN grown by MOCVD is 900 cm²/V's [2]. Recently high value of electron mobility above 1100 cm²/V's has been achieved for GaN films using MBE but these layers were grown on low dislocation density MOCVD GaN templates [3].

In this work nitride heterostructures were grown in specialized nitride STE3N MBE system (SemiTEq) using NH₃ as nitrogen precursor. The unique features of this system include substrate temperatures being extreme for typical MBE (up to 1200⁰C defined by IR pyrometer) as well as high N/III ratios (NH₃ flow up to 400 sccm).

An appropriate sequence of "thick" (200 nm) AlN layer grown on sapphire at high temperature of 1100-1150⁰C following by AlGa_{0.3}N transition layers (including superlattices) grown at 900-920⁰C results in high crystal quality of GaN layer grown on top. The STEM (scanning TEM) image of heterostructure consisting of AlN, SLS (superlattice), Al_{0.3}Ga_{0.7}N, gradient layer, Al_{0.1}Ga_{0.9}N and GaN layers, shows that dislocation density is 2-4·10¹⁰ cm⁻² in AlN, 4-6·10⁹ cm⁻² in AlGa_{0.3}N with Al content 30-10% grown after SLS and 9·10⁸-1·10⁹ cm⁻² in final GaN layer. For comparison, the dislocation density in GaN placed on top of a thin (10 nm) AlN buffer layer grown at moderate temperature (<900⁰C) lays in the end of 10¹⁰ cm⁻². In addition, it was found by TEM that the density of screw dislocations is ~2·10⁸ cm⁻², the density of edge dislocations is ~4·10⁸ cm⁻² and the density of mixed dislocations is ~4·10⁸ cm⁻².

Significant reduction of dislocation density has led to substantial electron mobility increase in active GaN layers. Maximum electron mobility in silicon doped 1.5 μm thick GaN layer is in the range 600-650 cm²/V's at carrier concentrations of (3-5)·10¹⁶ cm⁻³. The experimental values of mobility and dislocation density are in good agreement with calculations [4]. Additionally, it was found by AFM that surface rms is 2-4 nm for GaN that confirms a high quality of the material.

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Surface reconstruction affecting topography during metal-organic vapour phase epitaxy of nitrides

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For a long time the atomic arrangement (reconstruction) of nitride surfaces in metal-organic vapour phase epitaxy (MOVPE) was not considered important. This notion stems from GaN, where investigations by grazing X-ray scattering [1] and spectral ellipsometry [2] found a single (1x1) reconstruction above 700°C. Only by pulsing TMGa one could change the reconstruction [2].

In my contribution, I will show that the reconstruction is important for InGaN or AlN. Both material systems exhibit characteristic changes of the topography when changing growth conditions. For instance, InGaN with indium contents of 10% or higher exhibits monolayer high islands even after growth of 3 ML. By spectral ellipsometry we found the reconstruction changing during growth whenever a certain amount of TMIn was present. The two reconstructions trigger the formation of monolayer high islands [3].

The second system is AlN buffer growth on AlN (0001) / sapphire templates. High V/III ratios results in irregular surfaces and enhanced growth at screw dislocations. At medium V/III ratios a strong step-bunching occurs, while lower V/III ratios give smooth surface with evenly spaced steps. This was observed in three different types of MOVPE reactors (horizontal, CSS, and planetary), at growth temperatures between 1050°C and 1200°C.

Due to this range of temperatures, the occurrence of step-bunching of AlN (0001) cannot be limited by diffusion. Rather the topography results from three different reconstructions, that affect diffusion and the Schwoebel barrier (the energy for attachment at a step from the upper or lower terrace). Low V/III ratios likely form a metal ad-layer reconstruction, which is typical for nitrides. Such a reconstruction should enhance diffusion. However, it does not promote step-bunching, i.e. it has a positive Schwoebel barrier. At intermediate V/III ratios an Al-adatom structure might exist, which still has a long diffusion length. But this reconstruction has a negative Schwoebel barrier and causes step bunching. High V/III ratio might result in nitrogen-rich adatom structure, with much shorter diffusion lengths.

Thus in my contribution I will show that the reconstruction of nitrides can be important to understand the growth process of InGaN and AlN.

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Selective area growth of GaN on r-plane sapphire by MOCVD

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The use of non-polar GaN (11-20) allows avoiding polarization-induced electric field effects, which are presented in conventional III-N structures with (0001) orientation. Development of ELOG technology for such layers allows improving the quality of these structures by reducing the dislocation density. Usually, stripes for ELOG process are oriented perpendicular to GaN c-axis, which results in fast lateral growth in (0001) direction and low growth in (11-20) and (000-1) directions. Here we investigated the ELOG process peculiarities for other stripe orientations.

Stripe masks were fabricated on non-polar GaN (11-20), grown on the r-plane sapphire. Our photomask allowed us to investigate growth for different stripe orientation (the angles between the window and the c-axis were: 0°, 7.5°, 15°, 30°, 45°, 60°, 75°, 82.5°, 90°) and mask width between the stripes (5mkm, 10mkm, 20mkm, 40mkm).

For stripes oriented along (1-100) direction under high TMG flow and low NH₃/H₂ we observed the described above well-known results [1, 2]. Disorientation of the stripe by 7.5 degrees led to approximately symmetrical lateral overgrowth. This mode was also preserved for 15 degrees disorientation. However further increase in the angle led to significant decrease in the growth rate of one wing and increase in the growth rate of another one. This mode was preserved till the 90 degree, when the stripes acquired trapezoidal shape with low lateral overgrowth. Thus, at least two of three abrupt changes in the anisotropy of growth occur when the orientation of the stripe changes to low disorientated. We speculate that the density of the atomic steps on the side faces of the stripe impact on the growth rate of these faces. In particular, the (0001) face grows much faster than the (000-1) face, when the strip is oriented precisely perpendicular to the c-axis, due to the differences in their properties. However, at low disorientation the density of the steps on the (0001) and (000-1) faces increases dramatically, which leads to more efficient material embedding. Thus the growth rate becomes identical for both lateral directions.

For all the orientations of the stripe (except the precise orientation along the c axis) the possibility of a-GaN ELOG process was demonstrated.

For 45° stripe orientation more complicated double-cross ELOG technique was developed. The details will be presented.

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Various types of GaN/InGaN nanostructures grown by MOCVD on Si(111) substrate

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At the moment most of the device structures based on A₃B₅ compounds are grown using planarepitaxy, which leads to dislocations formation in the case of lattice-mismatched growth. For the heterostructures based on group III nitrides high density of threading dislocations is due to the mismatch of the lattice parameters of the growing material and the substrate. At the same time, there are reports showing that during the growth of structures with submicron lateral dimensions is possible to obtain defect-free nanostructures [1] In this work we developed methods of formation of defect-free nanowires using MOCVD and investigated their properties.

All the samples were fabricated on pre-grown AlN/Si(111) templates with a thickness of AlN 100 nm using MOCVD. The pulsed growth mode was used in which the group III and group V precursors were injected alternately with TMGa and NH₃ flow rates of 10 sccm and 0,002 slm, respectively, with a pulse rate of 13s. The growth temperature was 750C. In the most of experiments selective growth was promoted by pre-deposition of Au film with nominal thickness about 1 nm by conventional thermal evaporation resulted in ~10 nm in diameter Au islands formation as confirmed by atomic force microscopy.

The first kind of nanowires was about 350 nm in high and about 10 nm in diameter. The density of the nanowires was very high. The diffraction peaks from the (002) and (004) planes of the wurtzite- type hexagonal GaN NWs were observed, which indicated that the grown GaN NWs were preferentially oriented in the c-axis direction. For this kind of structures we found out growth temperature range in which nanowires growth is possible. The temperature range was 710-840C. Also we discovered that increasing in NH₃ flow, TMG flow, NH₃ step time and TMG step time led to polycrystalline growth between the nanowires and on the tops of them. So the NH₃/TMG parameters range is very narrow. For the second type of structures we added TMI during the initial stage of the growth. This resulted in the nanowires diameter increase up to 100 nm, which was caused by collecting indium droplets on the tops of nanowires. Also for this grow mode the lateral growth was observed, which may be due to In incorporation. For the next type of structures we deposited InGaN layer on the top of 100 nm-nanowires in the standard growth mode of planar InGaN. In this case the same oriented faceted pyramids were formed on the top of nanowires. For the last kind of structures we tried to promote three-dimensional growth without Au catalyst. For this purpose we introduced TMI into the reactor during all the growth time. This growth mode resulted in the uniform spherical structures with high density and clear AlN surface between them. XRD rocking curve of this structure showed peaks corresponded to In_{0,06}GaN and no metallic Ga peaks. All grown structures were investigated by scanning electron microscopy, photoluminescence and Raman spectroscopy and X-ray diffractometry. The results will be presented.

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Routes to Aluminium Gallium Nitride Buffer Layers for UV-LED Growth

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Ultra violet light emitting diodes (UV-LEDs) have many potential applications in biomedical engineering, and sterilisation, with the biggest potential market in the treatment of drinking water. However the efficiencies of UV-LEDs remains very low at 1-2 %, compared to over 70% for blue emitting GaN/InGaN LEDs. Reasons for this include high threading dislocation densities (TDDs) in the AlGaN buffer layers, low quantum efficiencies in $\text{Al}_x\text{Ga}_{1-x}\text{N}/\text{Al}_y\text{Ga}_{1-y}\text{N}$ quantum wells and the use of absorbing GaN p-type caps. In this study we compare routes towards achieving relaxed AlGaN buffers with low TDDs, grown by MOCVD on c-plane sapphire substrates, avoiding expensive bulk AlN substrates. AlGaN cannot be grown directly on sapphire, which necessitates that growth is based on an AlN or GaN template. If a GaN layer, this must be removed along with the sapphire substrate in a flip-chip processing geometry, but this is advantageous for UVLEDs anyway. This initial AlN or GaN layer must be followed by layers that relax the strain without creating threading dislocations, introducing cracks or that lead to surface roughening. We compare three different routes to growing high quality relaxed $\text{Al}_{0.5}\text{Ga}_{0.5}\text{N}$ buffers: using GaN/AlN superlattices on both AlN and on GaN substrates, and using low temperature AlN interlayers on a GaN substrate. Our GaN templates have a lower TDD compared to AlN: 002 FWHM are 250'' for GaN and 350'' for AlN, and 101 FWHM are 500'' for GaN and 1000'' for AlN.

AlGaN grown directly on GaN without any strain relief relaxes by creating screw type dislocations, with a 002 FWHM of 450'' and 101 FWHM of 1000''. Using a 100 period 0.25 nm GaN / 0.25 nm AlN superlattice prevents cracking in a 1 μm $\text{Al}_{0.5}\text{Ga}_{0.5}\text{N}$, but at the consequence of roughening (with values of 2nm for AlN, and 15 nm for AlGaN).

AlN/GaN superlattices grown on GaN will also similarly prevent AlGaN buffers from cracking, but relax by increasing surface roughness. Superlattices with a small period of 1 nm AlN / 1 nm GaN did not relax the AlGaN layer, whereas thicker 5 nm AlN / 5 nm GaN superlattices with a period of 5nm did relax, but by creating a large number of screw dislocations, the 002 FWHM was 800'' and 101 FWHM 900''.

We have found the best way to grow relaxed $\text{Al}_{0.5}\text{Ga}_{0.5}\text{N}$ buffers is to use multiple 10 nm thick AlN interlayers grown at the low temperature of 650°C, interspaced with 10 nm GaN layers. Using two interlayers gives a smooth AlGaN surface, with only moderate cracks around the wafer edge. 002 FWHM of 250'' and 101 FWHM of 1000'', show that only edge dislocations are created to enable relaxation. When more interlayers are used the crack density reduces further but more edge dislocations are created, whereas only using one resulted in the cracks penetrating further towards the wafer centre.

These buffer layers have been used to grow prototype polarisation-matched InAlN/AlGaN multiple quantum wells.

Influence of Sapphire Substrate Miscut Angle on Indium Content of MOVPE-grown InGaN Films

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Fabrication of green-light emitters, laser diodes requires the use of InGaN alloys with a high indium (In) content. The main barriers are inefficient indium incorporation during the metal-organic vapor-phase epitaxy (MOVPE) growth, resultant inhomogeneous broadening and the degradation of the quantum efficiency of the luminescence in green region. The problems are known as “green gap”. To overcome these problems, the enhancement effect of substrate miscut angle on In incorporation has been studied. However, there is only report of sapphire-substrate miscut angle less than 0.5°^[1]. In this study, we investigated the influences of much higher sapphire-substrate miscut angle from 0.4° upto 1.0° on the properties of MOVPE-grown InGaN films.

The *c*-plane sapphire substrates with different miscut angles (0.4, 0.6, 0.8 and 1.0°) toward *m*-direction were prepared. The GaN template and the InGaN films were grown by MOVPE. After the degreasing and the removal of the surface oxide by dipping into piranha solution, substrates were heated up for 5 min at 1040°C under hydrogen ambient in the reactor for thermal cleaning. GaN buffer layers and GaN templates were grown at 550°C and 1030°C, for 3.5 min and 180 min, respectively. Finally, InGaN films were successively grown at 800°C for 60 min. All the samples were grown simultaneously. The film thickness of GaN templates and InGaN films, measured by stylus profiler, are around 2.45 μm and 0.48 μm, respectively. The In content *x* of InGaN films were derived from X-ray diffraction (XRD) reciprocal-space maps (RSMs) of (105) diffractions using Poisson effect and Vegard's law. All the XRD measurements were performed using high-resolution X-ray diffractometer (Bruker-AXS, D8-Discover). Additionally, we measured XRD 2θ/ω profiles of symmetric plane (002) diffraction, the optical absorption, and the photoluminescence (PL). The In content *x* on the 0.4, 0.6, 0.8 and 1.0°-off substrate are 8.0, 8.0, 8.4 and 8.8 %. RSMs from all the samples indicate that the InGaN films are almost fully strained under in-plane compressive stress induced by GaN template. The 2θ of XRD 2θ/ω profile and the optical absorption edge energy of InGaN decreased with increasing the miscut angle of the substrate. These results well agree with the variation of In content *x* as determined from RSMs. The PL spectra exhibited multiple shoulder structures. However, these multiple peaks seen in the PL spectra must be attributed to the optical interference fringes due to multiple reflections within the air/InGaN film/GaN template system.

The enhancements of In incorporation in InGaN films with increasing the miscut angle of *c*-plane sapphire substrates were clearly observed, as well as the consistency in all the investigated properties such as the In contents, the optical absorption edge energy and the PL peak positions. The enhancement of In incorporation with increasing the miscut angle can be attributed to the increase in the step density, since dangling bonds at step edges suppress the detachment of In atoms.

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Growth of GaN layer with high crystallinity on free-standing GaN substrate using Ga₂O as Ga source

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Recently, commercially available GaN substrates are produced wafer by wafer, which complicates the production process and increases the production cost. One of the most effective ways for commercial production of GaN substrate is a development of bulk growth with subsequent wafer slicing. The important features required for the bulk GaN growth method are high growth rate, high crystallinity, high purity, and long-term growth. The growth method of GaN crystal using Ga₂O allows long-term growth, in principle [1]. In this method, Ga₂O vapor is produced by the reduction of Ga₂O₃ powder with H₂. Then, Ga₂O react with NH₃ and synthesize GaN. In our previous report, we reported high temperature growth (~1250°C) can allow to increase growth rate of GaN layer with low oxygen concentration in polar and non-polar epitaxial layers [2,3]. In this report, we tried to grow polar and non-polar GaN layers with high crystallinity on free-standing GaN substrate in order to improve crystallinity.

Commercially available *c*- and *a*-plane free-standing GaN substrates are used as seed substrates. Full Width at Half Maximum (FWHM) of X-ray rocking curve of *c*- and *a*-GaN substrates were 115-131 arcsec and 23-43 arcsec with incident X-ray parallel to *c*-axis and 25-63 arcsec with the incident X-ray perpendicular to *c*-axis, respectively. Commercially available Ga₂O₃ (4N) was used as starting material and was reduced by hydrogen gas (6N). NH₃ (3N) and N₂ (5N) gasses were used as the nitrogen source and carrier gas, respectively. Epitaxial growth was performed in a home-made horizontal quartz tube consisting of the source and the growth zones. A tungsten tube containing Ga₂O₃ powder set in a molybdenum boat was placed in the source zone. The seed substrate was placed in the growth zone. The source and the growth zones were maintained 840-1000°C and 1200-1250°C, respectively, for the growth period 1hr.

In case of *c*-plane, the number of pits consisting of (10-11) or (10-12) facet was increased and the FWHM of GaN (0002) X-ray rocking curve of the epitaxial layer was decreased with increasing the growth rate. Compared with the FWHM of the seed substrate, that of epitaxial layer was decreased. The *c*-GaN epitaxial layer with the growth rate of 39 μm/h and the FWHM of 74 arcsec was obtained. In case of *a*-plane, smooth epitaxial layers were obtained. The FWHM of GaN (11-20) of epitaxial layer was almost the same as that of the seed substrate. The *a*-GaN epitaxial layer with the growth rate of 46 μm/h and the FWHM of 29 arcsec and 24 arcsec with the incident X-ray parallel to and perpendicular to *c*-axis was obtained, respectively.

These results indicate that growth on high quality free-standing GaN substrate can be an effective way to obtain polar and non-polar GaN crystal with high growth rate and high crystallinity.

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Effect of Oxygen Partial Pressure on the Growth of Single-Crystalline Aluminum Nitride Layer using Liquid Phase Epitaxy Technique

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Aluminum nitride is a promising substrate material for AlGaIn-based deep ultraviolet light emitting diodes (UV-LED) because of its UV transparency and small lattice mismatches with AlGaIn. We have been developing a new liquid phase epitaxy method using Ga-Al flux to obtain high quality AlN layer on nitrided sapphire substrates [1-3]. Full width at half maximum values of (0002) and (10-12) X-ray rocking curves of the LPE AlN layer are 50 and 590 arcsec, respectively.

In this study, we have investigated effects of oxygen partial pressure P_{O_2} on the LPE AlN growth with controlling P_{O_2} in injecting N_2 gas. We found that the LPE AlN layer successfully grew at higher P_{O_2} (10^{-1} Pa), however, the growth rate significantly decreased at lower P_{O_2} (10^{-12} Pa).

We studied the polarity of AlN layers using convergent-beam electron diffraction (CBED) [4], and it was clarified that the LPE AlN layer has Al-polarity even though the nitrided sapphire layer has N-polarity. Thus, polarity inversion occurred at the interface between nitrided sapphire layer and LPE layer.

From the results of polarity determination and growth experiment using different P_{O_2} in injecting N_2 gas described above, we consider the polarity inversion was caused by the presence of thin oxygen-containing interlayer between nitrided sapphire layer and LPE layer. This idea is supported by Wong *et al.* who reported the polarity inversion of N-polar GaN using an aluminum oxide interlayer for MBE growth of GaN [5].

The nitrided sapphire layer having N-polarity dissolved into the flux under lower P_{O_2} (10^{-12} Pa) condition, because no oxygen-containing interlayer formed during the LPE process. This is thought to be the cause of much lower growth rate obtained for the LPE process under lower P_{O_2} . In contrast, in the case of higher P_{O_2} (10^{-1} Pa) condition, polarity inversion occurred because of formation of oxygen-containing interlayer, and therefore, the surface of nitrided sapphire layer changed from N-polarity to chemically stable Al-polarity, which enables the successful homoepitaxial growth of AlN layer. Thus, oxygen partial pressure in injecting N_2 gas has an important role to grow AlN layer on the nitrided sapphire substrates using the LPE process.

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Dramatic improvement in the crystalline quality of AlN grown on thermally-nitrided sapphire substrates by pulsed sputtering deposition

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AlN is an attractive substrate material for AlGaIn based ultraviolet (UV) light emitting devices because of its transparency and small lattice mismatches. Recently, it has been reported that the surface nitridation of sapphire leads to the formation of high-quality AlN ultrathin templates by precisely controlling of the driving force for the thermal nitridation reaction in carbon-saturated N₂-CO gas mixture.[1,2] However, the threading dislocation density in this film is still high with the order of 10⁹ cm⁻². The reduction in the dislocation densities by the subsequent epitaxial growth is highly required for the fabrication of high performance optical devices on this template.

In this study, we demonstrate the epitaxial growth of high-quality AlN films on the thermally nitrided sapphire substrates by pulsed sputtering deposition (PSD). We report on that the precise control of growth conditions results in the dramatic improvement in the crystalline quality of AlN.

Growth of AlN films was performed by PSD. The substrate temperature was varied from 900 to 950 °C. The sputtering of an Al metal target was carried out in argon and nitrogen gaseous ambient and the pulsed dc power was applied at 100-200 W. The Al-flux and N-flux were varied from 1.6 to 2.4×10¹⁴ atoms/cm²s, respectively, which were estimated from the growth rate at low temperature. The surface stoichiometry was monitored by *in-situ* reflectivity measurements and RHEED. The growth regimes such as N-rich or Al-rich were also identified by AFM observations.

Under N-rich growth condition, the degradation of the crystalline quality and serious surface roughening were observed, which spoiled the advantages of high-quality AlN templates fabricated by nitriding sapphire. On the other hands, under slightly Al-rich condition, the FWHM values of x-ray rocking curves for 1-102 diffraction decreased with the increase in the film thickness. For the sample with the film thickness of 400 nm, the FWHM values for 0002 and 10-12 were as low as 147 and 339 arcsec, respectively. These values were comparable with those of AlN grown on sapphire by sophisticated MOVPE growth techniques. [3,4] This improvement was probably explained by the enhancement of surface migration of Al, which was realized by pulsed dc sputtering operation and the precise control of surface stoichiometry. These results indicate that the combination of PSD and thermally nitrided sapphire is quite promising growth technique for fabrication of future UV light emitting devices.

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A study of GaN regrowth on the micro-faceted GaN template formed by in-situ HCl etching

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HVPE has absorbed more interesting due to its high growth rate. Despite the successful fabrication of free-standing GaN substrate using HVPE by several groups [1], some critical problems, such as the high dislocation density, cracking and bowing, are still difficult to be solved. In this paper, in-situ HCl etching method is provided for the growth of high-quality and crack-free GaN thick films.

The micro-faceted templates were formed by in-situ HCl etching in a vertical-type HVPE system under high temperature 1050 °C. First, the morphology of GaN etched in HCl atmosphere is investigated. A large number of hexagonal pits on the surface are observed. It is found that there were two different kinds of hexagonal pits sizes on the surface and the density of the pits would increase with the etching time adding to 7 minutes. Then, thin GaN flim has been grown on micro-faceted template for 3 minutes. A lot of cylinder structures can be observed on the surface, sizes of which are increasing from several microns to dozens of microns with different regrowth time. Finally, the etched surface will become smooth for long time regrowth, and the coalescent of the cylinder structures is a key to the improvement of the crystal quality and the release of the strain in the GaN films. A simple mechanism is given to explain it. Meanwhile, cross-sectional structure of GaN thick films which were in-situ etched for six times were investigated by CL. It is clear that this etched GaN has six sublayers in the film. Full size of 2 inch crack-free GaN films with thickness about 300 μm have also been obtained by in-situ etching for several times during the growth.

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Step bunching and meandering induced by natural step flow and its role in the dynamics of crystal growth

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Crystal growth dynamics and its relation to formation of various geometric patterns remains a subject of continuous interests of many researchers. At its foundation, crystal growth geometry was supposed to follow simple scheme devised by Burton Cabrera and Frank (BCF). Additional phenomena were added to the picture either emerging during Monte Carlo simulations or observed in the real growth experiments. It was noticed that parallel steps have tendency to create step trains, in accordance to prediction of kinematic step train theory. In parallel to the step motion instabilities, the structure of the steps could be affected by long range fluctuations. The instability phenomena, denoted as step meandering, were observed during growth of various type crystals. The analytical approaches intensively developed during last decades are able to provide trends and scaling behavior of the meandering step system. Yet the critical assessment of these theories requires careful analysis of the growth experiments, and parallel Monte Carlo simulations. Recently we have proposed the model which was able to recover basic features of the growth of GaN layers by metalorganic vapor phase epitaxy (MOVPE) [1,2] It was shown that the step anisotropy could be obtained within model based on four-body interaction.

Growth of gallium nitride on GaN(0001) surface, modeled by Monte Carlo method is analyzed under various external parameters. Simulated growth is conducted in N-rich conditions, hence it is controlled by Ga atoms surface diffusion. Dominating four-body interactions of Ga atoms cause step flow anisotropy. Kinetic Monte Carlo simulations show that parallel steps with periodic boundary conditions form double terrace structures. Step meandering during growth of GaN (0001) surface is studied. It appears that at relatively high temperatures and low fluxes steps move regularly as straight, parallel lines. For wide range of fluxes and temperatures step meandering happens even if Schwoebel barrier is set to zero. We show that depending on the initial surface parameters, two different scenarios of step meandering are realized. In both these regimes meandering has different character as a function of external flux and time. Additionally it is shown how step meandering patterns are destroyed by too high Schwoebel barrier. On studying sublimated GaN surface we show emergence of step bunches at the surface within the same crystal model. We study step bunching process at different condition. We show the mechanism of step pairing, step meandering and bunching in the systems with no step edge anisotropy. The only reason for step instability is step flow during crystal surface dynamics process. All structures, emerging in the simulations, have their corresponding cases in the experimental results.

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Growth of AlGaN on sapphire using AlN/GaN superlattices as strain relaxing layers

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There are many applications for ultra violet light emitting diodes (UV-LEDs), the largest is the sterilisation of drinking water but there are many others such as in new medical treatments and in improving the ease and efficiency of biological assays. UV-LEDs normally require a relatively thick *n*-doped AlGaN buffer layer, with the composition depending on the desired output wavelength. As AlGaN is not usually grown on sapphire directly one needs to relax an AlGaN layer grown on AlN or GaN without roughening or creating excess threading dislocations. In the latter case the substrate and GaN-containing strain reducing stacks will need to be removed by a flip chip process, but this geometry has other advantages for UV-LEDs such as minimising the effect of absorbing *p*-type GaN cap (it occurs underneath of LED chip) and having a heat sink closer to the active region.

We have studied four AlN/GaN superlattices grown on AlN templates. All were capped with 1 μm of AlGaN.

The three GaN/AlN superlattices grown on GaN templates had the same total thickness of 400 nm but different periodicities of 10 nm, 4 nm and 2 nm. The 5 nm/5 nm superlattice fissured into microcracks during growth which were overgrown, but still leading to a very rough surface and creating screw type dislocations. In-situ optical monitoring showed significant roughening during the 2 nm/2 nm superlattice growth and the surface was smoothed somewhat during the AlGaN growth. There is no evidence of surface roughening during 1 nm/1 nm superlattice growth, but it did not cause the AlGaN to relax. A 100 period 0.25 nm/0.25 nm GaN/AlN superlattice like in the case of 1 nm/1 nm one prevented the subsequent AlGaN from cracking but led to its surface roughening.

In conclusion, GaN/AlN superlattices can help in crack preventing but none of these studied here achieved smooth top AlGaN surfaces.

AlGaN/AlN quantum dots for UV light emitters

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The development of solid-state ultraviolet (UV) emitters would imply a mayor advance for applications like water/air purification, bio-detection, or phototherapy. In the recent years, AlGaN-based UV light-emitting diodes (LEDs) have appeared as promising candidates to replace mercury lamps due to their advantages of compactness, portability, long lifetime and environmental friendliness. However, electrically-driven UV LEDs face the challenges of *p*-type doping and contacting high-Al-content AlGaN layers, which results in extremely low external quantum efficiency (EQE < 5% for pulsed operation at 280 nm wavelength). Electron-pumped UV (EPUV) sources incorporating a miniaturized electron source have been introduced as an alternative approach to circumvent the *p*-doping issue. A key requirement for EPUV devices is an active media with high internal quantum efficiency (IQE) at room temperature. This fact motivates the use of AlGaN/AlN quantum dots (QDs), where the three-dimensional (3D) carrier confinement grants certain insensitivity to non-radiative recombination processes.

In this work, we present the growth and properties of two types of AlGaN/AlN QDs, namely strain-induced Strankski-Krastanow (SK) QDs and nanodisks (NDs) created by nanowire (NW) heterostructuring. All the samples were synthesized by plasma-assisted molecular beam epitaxy. The assessment and comparison of these nanostructures is relevant for the EPUV application: Nominally both approaches present similar 3D carrier confinement, but SK-QDs provide a better QD size uniformity along the growth axis, whereas the ND geometry ensures better light extraction efficiency.

Regarding SK-QDs, 100 stacks of AlGaN/AlN QDs were deposited on (0001) AlN templates. The dots were grown under N-rich condition to activate the SK growth mode while the AlN barriers were deposited in metal-rich regime to flatten the surface. Short growth interruptions and relatively low substrate temperature were used to obtain QD density in the range of 10^{11} - 10^{12} cm⁻², with QD height ~ 1-1.5 nm and base diameter < 10 nm. By varying Al/Ga flux ratio, the emission wavelength can be tuned in the range of 245-350 nm at room temperature, keeping IQE values around 0.5 in all the spectral range.

The ND samples under study consist of 15-30 stacks of AlGaN/AlN heterostructures which were deposited on the top of GaN NWs on Si(111). The abrupt hero-interfaces and the ND size control along the NW structure are illustrated in Fig. 2(b). The ND diameter is controlled by the wire diameter while their height is determined by the heterostructure thickness. The optical studies reveal room-temperature IQE around 0.3 in the 280-350 nm spectral range. The emission spectra from the AlGaN/AlN NDs show larger linewidth than that of SK-QDs or of GaN/AlGaN NDs, which could be attributed their larger size and chemical composition inhomogeneities.

Growth of InN Nanostructures by Droplet Epitaxy Techniques

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The InN material has been recently attracted much attention, largely due to its narrow direct bandgap energy of ~0.65-0.85 eV. InN quantum dots (QDs) are highly interesting owing to nanometer-scale charge carrier confinement in all three spatial dimensions. This gives rise to quantized energies of the QDs leading to applications such as lasers, photo-detectors, light emitting diodes and THz generation. To fabricate InN dots, the stranski–krastanow (SK) growth mode and recently the droplet epitaxy (DE) technique has been utilized. In DE technique, Indium (In) droplets are exposed to a subsequent nitrogen (N) plasma beam, to convert the In droplets into InN dots. Compared with other growth techniques, size and density control of the dots are relatively easier in droplet epitaxy, by controlling by the amount of the supplied metals. InN QDs were fabricated on silicon nitride/Si (111) substrate by droplet epitaxy using plasma-assisted MBE system. Variation of the growth parameters, such as growth temperature and deposition time, allowed us to control the characteristic size and density of the QDs. As the growth temperature was increased from 100°C to 300°C, an enlargement of QD size and a drop in dot density were observed, which was led by the limitation of surface diffusion of adatoms with the limited thermal energy. The chemical bonding configurations of InN QDs were examined by X-ray photo-electron spectroscopy. Fourier transform infrared spectrum of the deposited InN QDs shows the presence of In–N bond. Photoluminescence measurement shows a slight blue shift compared to the bulk InN, arising from size dependent quantum confinement effect. The interdigitated electrode pattern was created and current–voltage (*I-V*) characteristics of InN QDs were studied in a metal–semiconductor–metal configuration in the temperature range of 80–300 K.

UV Photoresponse of GaN Nanodots grown by Molecular Beam Epitaxy

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Group III-nitride materials have been extensively investigated due to their applications in light-emitting diodes, laser diodes, and photodetectors (PDs). Ultraviolet (UV) PDs are important devices that can be used in various civilian as well as military applications such as space communications, ozone-layer monitoring, and flame detection. The wide direct bandgap and the excellent chemical and thermal stability of GaN make it particularly suitable for extreme environmental applications of UV PDs. The present work explores the electrical transport and ultraviolet photoresponse properties of GaN nanodots (NDs) grown by plasma-assisted molecular beam epitaxy. Single-crystalline wurtzite structure of GaN NDs is verified by the X-ray diffraction and transmission electron microscopy. The interdigitated electrode pattern was created and current–voltage (I–V) characteristics of GaN NDs were studied in a metal–semiconductor–metal configuration. The dark I–V characteristics of lateral grown GaN NDs were obeyed the Frenkel-Poole emission model. It was also found that dynamic UV response of the device was stable and reproducible with an on/off current contrast ratio of around 10. The responsivity of the detectors is found to be 330 A/W with an external quantum efficiency of 1100%, for an incident wavelength of 360 nm at 3 V.

Patterning of Pd and AlGaN/GaN superlattice for the fabrication of blue laser diodes

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Currently, laser displays ranging from large-size laser TV to mobile projectors, are commercially available or due to appear on the market [1]. In order to achieve a mobile projectors, the semiconductor laser diodes should be used as a laser source due to their size and weight.

In this presentation, the continuous etch characteristics of Pd and AlGaN/GaN superlattice (100 periods) for the fabrication of blue laser diodes were investigated by using inductively coupled CHF₃ and Cl₂ –based plasma.

The GaN laser diode sample were grown on the sapphire(0001) substrate using a metal organic chemical vapor deposition system. A Si-doped GaN layer was grown on the substrate, followed by growth of LD structures, including the active layers of InGaN/GaN quantum well and barriers layer, as shown in other literature [2], and the palladium was used as a p-type ohmic contact metal.

Up to date, the etch characteristics of Pd was not fully understood, because the suitable gas to form a volatile etch-product was not known due to the chemical inertness of Pd.

While when the Cl₂/Ar plasma were used in order to etch the AlGaN/GaN superlattice (SL), the etch rate was similar as that of n-GaN, the Cl₂/CHF₃ plasma shows decreased etch rate, compared with that of Cl₂/Ar plasma, especially for AlGaN/GaN superlattice. It was also found that the Pd which is deposited on top of the superlattice couldn't be etched with Cl₂/Ar plasma indicating that the etching step should be separated into 2 steps for the Pd etching and the superlattice etching, respectively. So we used 2-step process including separate Pd and SL etching by using Cl₂/CHF₃ and Cl₂/Ar, respectively. EDX results shows that the etched surface is a GaN waveguide, free from the Al, indicating the SLs were fully removed by etching. Furthermore, the optical and electrical properties will be also investigated in this presentation.

In summary, Pd/AlGaN/GaN SLs were successfully etched exploiting noble 2-step etching processes.

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InGaN quantum dot formation mechanism on hexagonal GaN/InGaN/GaN pyramids

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Growing InGaN quantum dots (QDs) at the apex of hexagonal GaN pyramids is an elegant approach to achieve a deterministic positioning of QDs. Despite similar synthesis procedures, the optical properties of the QDs reported in the literature vary drastically. QDs tend to exhibit either narrow ($\sim\text{\AA}$) or broad ($\sim 10\text{-}50\text{ nm}$) emission lines in the micro photo luminescence (μPL) spectra. By coupled micro-structural and optical investigations, the QDs giving rise to narrow emission properties were concluded to nucleate in association to a (0001) facet at the apex of the pyramid. In addition, single-exciton properties of pyramid grown QDs will be discussed.

The InGaN QDs were fabricated on top of site-controlled GaN hexagonal pyramids by a hot-wall MOCVD process and selective area growth. The pyramids had six equivalent {10-11} sidewalls, upon which one InGaN quantum well was grown, and subsequently a thin GaN cap layer was deposited. The edges of the GaN pyramids were about $1.7\mu\text{m}$ in width and $3.2\mu\text{m}$ in height.

The apex microstructure of two FIB cut pyramids were investigated by cross-sectional (S)TEM imaging and (S)TEM-EDX mapping of the indium-K line. The apex of pyramid 1 exhibits nearly homogenous Z-contrast and –indium-K signal indicating the formation of a crescent shaped InGaN QW composed of an apparently homogenous InGaN alloy. The indium-K map of pyramid 2 revealed the existence of a (0001) planar oriented InGaN QW located at the (0001) facet of the pyramid. The Z-contrast of pyramid 2 is inhomogeneous at the InGaN/GaN interface with two prominent indium-rich InGaN clusters clearly visible. Pyramid 2 exhibit sharp emission peaks ($\text{FWHM} \ll 1\text{ meV}$) in the μPL spectrum. No sharp features were observed for pyramid 1. Accordingly we suggest the sharp emission originate from the indium-rich InGaN clusters observed in pyramid 2. The hypothesis is further supported by additional CL and AFM measurements.

Controlled growth of hexagonal GaN pyramids by hot-wall MOCVD

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Selective area growth (SAG) by metal organic chemical vapor deposition (MOCVD) has been proven to be an efficient procedure for fabrication of site-controlled three dimensional GaN nanostructures. SAG experiments performed on (0001) oriented GaN templates with circular SiN apertures and most often result in hexagonal GaN pyramids with dominating semi-polar {1-101} facets. However, bi-facets such as non-polar {1-100}, and semi-polar {1-102} –facets are often observed on these pyramids. In this paper, the pyramid facet formations are investigated. By the use of proper growth conditions, the bi-surfaces were completely eliminated resulting in highly uniform pyramid arrays.

A (0001) oriented GaN epitaxial film was used as template for the hexagonal GaN pyramids. A 30 nm thick SiN layer was deposited by plasma-enhanced CVD. Subsequently, photolithography was adopted to transfer the pattern onto the SiN covered substrate, followed by inductively coupled plasma etching to open holes in the SiN mask layer. After chemical cleaning, the templates were placed into a hot-wall MOCVD reactor for hexagonal GaN pyramid growth.

Scanning electron microscope (SEM) images showed the successful growth of hexagonal pyramid with visible {1-101}, {1-102}, and {1-100} facets. By systematic investigation of the V/III –ratio, growth temperature, NH₃ flow, and growth time, we concluded that {1-100} facets formed in gallium rich growth conditions. According to earlier performed DFT calculations [1], gallium dimers preferentially form on {1-100} surfaces in gallium rich conditions. Such dimer formation reduces surface energy, and according to the Wulff theory of crystal growth appears the low energy surfaces on the post growth SEM images. Similarly {1-102} surfaces were removed in NH₃ rich conditions. We believe the removal of the {1-102} facets is caused by a hydrogen passivation layer [2] formed at the {1-101} surfaces. Highly uniform pyramid arrays were grown under optimized conditions.

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InGaN light-emitting diodes with the complex air structures with combination of triangular prisms and cones

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The effect of the combination of triangular prisms and cones as air-void structures arrays on the enhancement of light extraction efficiency (LEE) of InGaN light-emitting diodes (LEDs) is investigated. The arrays embedded at the sapphire/GaN interface act as light reflectors and refractors, and thereby improve the light output power due to the redirection of light into escape cones on both the front and back sides of the LED. Enhancement in radiometric power as high as 174% and far-field angle as low as 125° are realized with a compact arrangement of arrays compared with that of a conventional LED made under an injection current of 20 mA.

To analyze the LED structure, geometrical data for simple planar LED is presented. First, the sapphire substrate with size 650x220x100 μm is used. 4-μm-thick layer of Si-doped *n*-type GaN, five-period MQWs of InGaN/GaN pairs for a 460 nm emission and 110 nm *p*-GaN are subsequently placed, and finally, 300 nm indium titanium oxide (ITO) with refractive index of (1.9) is positioned. The surface source with Lambertian intensity distribution and 100 000 rays were used as an emitter to originate from the multiquantum well active layer. The size of the source matches the lens size of the LED. After simulation it was found the extraction efficiency of conventional planar LED is 15.9 %. Analysis of 3D air embedded structures can be provided by adding a bulb textures in the bottom of GaN.

From experimental point of view, it is very difficult to realize 3D void structures in GaN-sapphire interface practically, only using the method of growth-etching air voids-regrowth. So we propose more realistic one-step etching method of the round SiO₂ patterns connected by linear stripes. After wet etching process, the conical air structure under round pattern and prism structure under linear stripe are formed.

Such structures were analyzed with respect to distance/size parameter and also with various sizes of triangle prism. Results show that the size of prism channel should be minimal, size of cone – maximal with maximum fill-factor. Such structure allows improving the output power 2.7 times larger than planar one. Additional improving of previous structure can be realized using air prism and truncated cone structure like trapezoidal array.

In summary, the effect of combined cone and triangle prism arrays on the enhancement of LEE of InGaN LEDs has been systematically investigated. Using a one-step wet-etching process, well-aligned arrays have been formed in GaN. The optical output power was significantly improved due to light redirection toward front and back sides of the device. Using this approach, the radiometric power is enhanced by 174%, as compared with the conventional LED. This study ensures that embedded air-voids with optimal geometrical shape and wet etching technique would be effective in improving the optical performance of InGaN LEDs.

Optical Polarization Properties of a-plane GaN Light Emitting Diodes by Controlled Integration of Silica Nano-spheres

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The difficulties of nonpolar a-plane GaN growth on r-plane sapphire lies in the anisotropic planar growth rates, which results in undulated or trenced surface feature, large density of threading dislocations and stacking faults. In order to improve the material quality of nonpolar a-plane GaN by a simple process, we propose a controlled integration of silica nano-spheres (CIS) into the a-plane GaN epitaxial layer grown on r-plane sapphire by metal-organic chemical vapor deposition.

In addition to the crystal quality improvement by silica nano-spheres, we found that light emitted from CIS a-plane GaN LED has the reduced polarization property. To confirm the reduced polarization property by silica nano-spheres, we experimentally measured the polarization ratio of top-emitted light of CIS a-plane GaN LED and performed three dimensional (3-D) finite-difference time-domain (FDTD) simulations.

First, we obtained the polarization ratio from top-emitted light of CIS a-plane GaN LED by measuring electroluminescence (EL) intensity on polarizer angle. Polarization ratios of the CIS a-plane LED and the reference sample without silica nano-sphere were 0.27 and 0.59, respectively. Polarization ratio of CIS a-plane LED decreased by 0.32, compared to the value of a-plane LED without silica spheres.

In order to confirm the effects of polarization conversion by silica nano-spheres, we performed the 3-dimensional finite-difference time-domain (FDTD) simulation and polarization ratio of a-plane GaN LED with silica nano-spheres was calculated. We assumed a simple structure made of a GaN layer (refractive index: 2.43) with height of 1 μm on sapphire substrate. A layer of periodic silica nano-spheres (refractive index: 1.43) with diameter of 250 nm was arranged on the sapphire substrate. Surface coverage of silica nano-spheres was varied from 0 to 44.2 %. A perfectly matched layer (PML) boundary condition was employed as absorbing boundary at top and bottom site of simulation region and periodic boundary condition was employed along the four lateral boundaries to ignore the finite size of simulation model. The calculated results show that as the surface coverage increased from 0 to 44.2 %, the polarization ratio was decreased from 1.0 to 0.30. Since each silica nano-sphere could act as a scattering center which changes direction of oscillation in electromagnetic wave, polarization ratio decreases with increased surface coverage by silica nano-spheres. Calculated polarization ratio is similar to the experimental result for the sample with surface coverage of around 40%. More detailed results on light polarization of CIS a-plane GaN LED will be reported.

Layer thickness dependent electrical properties of MOCVD grown GaN layers

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Most of gallium nitride based device structures are grown heteroepitaxially, i.e. on sapphire, SiC, etc. Mismatch of lattice parameters and thermal expansion coefficient between substrate and GaN generates unacceptable density of defects. It limits the device performance and operating lifetime. Electrical properties of undoped GaN depending on layer thickness was studied and is presented in this work.

Set of GaN layers of different thickness were grown by low-pressure metalorganic chemical vapour deposition (MOCVD) technology using a 3x2" closed-coupled showerhead reactor. c-plane sapphire with a miscut of 0.25° towards m-axis was used as a substrate. Growth conditions were kept same for all samples investigated. The thickness of the grown layers varied from 2 to 25 μm.

Light-induced transient grating technique was applied to determine lifetime and diffusivity of excited charge carriers at temperatures from 300 K down to 10 K. Ambipolar carrier plasma with depth of about 0.1 μm ($\alpha = 10^5 \text{ cm}^{-1}$) was created after interband excitation (355 nm, picosecond Nd:YAG laser). Equilibrium electron concentration and mobility at room temperature were estimated from Hall measurements. Rocking curves (i.e. ω scans) for (002) and (302) reflections were measured in order to estimated screw and edge dislocation density.

We found out clear dependence of carrier lifetime and equilibrium carrier density on layer thickness. Carrier lifetime gradually increased with increasing thickness - from 0.7 ns for thin layers (~2.5 μm) up to 3.4 ns for thick ones (25 μm). Respectively the equilibrium carrier density decreased from $1.3 \times 10^{18} \text{ cm}^{-3}$ down to $2.9 \times 10^{16} \text{ cm}^{-3}$. Better photoelectric properties of thick layers are governed by reduced dislocation density which monotonically decreased with increasing sample thickness from $1.3 \times 10^9 \text{ cm}^{-2}$ down to $4.5 \times 10^8 \text{ cm}^{-2}$ at 25 μm thick sample.

The interplay of radiative and nonradiative recombination was investigated by temperature dependent carrier lifetime measurement. The lifetime of low-defected thick samples exhibited a classical dependence of $\tau \propto T^{3/2}$ in 10-100K range, showing that dominant carrier recombination mechanism is bimolecular (radiative). Weaker temperature dependence ($\tau \propto T^{1/2}$) for thin layers in 10-300 K range was attributed to impact of nonradiative recombination via defects. The increase of stimulate emission threshold for thicker layers is discussed.

It was found that carrier mobility is almost constant in all samples. Equilibrium carrier mobility was about $\mu_e = 170 \div 220 \text{ cm}^2/\text{V}\cdot\text{s}$, while ambipolar mobility of excess carriers was $\mu_a \approx 2\mu_h = 70 \text{ cm}^2/\text{V}\cdot\text{s}$. Mobility versus temperature dependence, $\mu_a \propto T^{-3/2}$, confirmed that the main carrier scattering mechanism is intrinsic one by acoustic phonons. This explains why layers with different defect density have similar mobility values.

Resistance formation mechanisms for contacts to trinitride heterostructures with high dislocation density

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We discuss a formation mechanism for ohmic contacts to *n*-GaN and *n*-AlN by exploiting the results of measurements of temperature dependences of the contact resistivity ρ_c . The corresponding curves involved three nontrivial portions, namely, (i) saturation portion (in the 100–150 K temperature range), (ii) activation portion (in the 150–200 K temperature range), with activation energy ≤ 100 meV, and (iii) portion of very weak temperature dependence (at temperatures > 200 K). The portions of (ii) and (iii) types were found for contacts to *n*-AlN in the 100–375 K temperature range. The GaN and AlN epitaxial layers with dislocation density $> 10^9$ cm⁻² were obtained with chloride vapor-phase epitaxy. For comparison the $\rho_c(T)$ dependences for ohmic contacts to *n-n*⁺-GaAs were studied too. It was shown that a necessary condition for realization of the above dependences in the saturation portion is either very heavy doping in the near-contact region or formation of a region with high concentrations of donors and structural defects in the course of ohmic contacts making.

Except for [1], there were no discussions of such $\rho_c(T)$ curves earlier. The explanation for behavior of $\rho_c(T)$ dependence in the 10–360 K temperature range for ohmic contacts to *n*-GaAs was given in [1]. It reduced to appearance of a heterojunction in the GaAs near-contact region in the course of contact making, with band offset in the heterostructure formed by GaAs and ternary metal–GaAs phase serving as barrier. In that case, however, the range of ρ_c low-temperature saturation is very narrow (10–40 K), while in our experiment (*n*-GaAs with close doping level) the range is more extended (up to 250 K). A possible explanation for the dependences obtained may be as follows. According to the model proposed by us, the portion (i) may be related to current flow between dislocations, while the portions (ii) and (iii) – to current flow through the metal shunts associated with dislocations. To reconcile the data on presence of a heavily doped layer in the semiconductor near-surface region with rather high ρ_c values, one should assume presence of a dielectric gap with sufficiently small transmission factor at the metal–semiconductor interface.

The run of the portion (ii) of $\rho_c(T)$ curve seems to be explained within the theory of conductivity of disordered media. As to the portion (iii), we believe that it is due to the mechanism of current flow limitation with diffusion supply of electrons to the dislocation ends. The results obtained make it possible to draw conclusions concerning specific character of the physical mechanisms responsible for current flow in ohmic contacts to wide-gap semiconductors with high concentration of structural defects.

[1] Contacts to Semiconductors. Fundamentals and Technology / Ed. by L.J. Brillson (Noyes. Publ., USA, 1993)

Ohmic and Schottky contacts to AlGa_xN layers with high Al content for deep ultraviolet optoelectronics

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AlGa_xN material system with the maximum band gap energy of 6.1 eV allows to manufacture ultraviolet (UV) optoelectronic devices working within a wide UV-A,B,C spectral range with a minimum wavelength of $\lambda=210$ nm. However, fabrication of the low-resistance ohmic contact to Al_xGa_{1-x}N:Si with a high Al content ($x > 0.5$) is a difficult and actual task [1]. There still exist many problems also with Schottky-barrier contacts to undoped Al_xGa_{1-x}N layers, necessary for fabrication of solar-blind photodetectors. This paper reports on the achievement of low resistance ohmic contacts to n-Al_xGa_{1-x}N:Si layers with Al-content $x \sim 0.6$ by using various metallic contacts and their treatment. In addition, the initial results on development of the Schottky barriers in Metal-Al_xGa_{1-x}N ($x=0.08-0.7$) structures are reported.

The n-type Al_xGa_{1-x}N:Si ($x = 0.5 - 0.6$) and undoped Al_xGa_{1-x}N ($x=0.08-0.7$) epitaxial layers with thickness of 1 μm were grown by PA MBE on c-sapphire substrates. The thermal vacuum evaporation of different metals (Ti, Au, Ni, Ag, In, Al) was used to deposit different single- and double-layer contacts onto the AlGa_xN layer surface cleaned preliminary with organic solvents. The samples were annealed immediately after the contact deposition in the same vacuum conditions ($<10^{-6}$ Torr) of a metal deposition chamber. The layers were characterized by Hall measurements in a Van der Pauw geometry using soldered indium contacts. The samples with the different contacts were studied by using the transmission line method (TLM), current-voltage characteristics and photo-response spectra measurements.

The Al_xGa_{1-x}N:Si ($x=0.5-0.6$) layers demonstrated the electron concentration up to $n=1.7 \cdot 10^{19} \text{ cm}^{-3}$ and mobility of $\mu \sim 18 \text{ cm}^2/\text{Vs}^{-1}$ while the undoped samples revealed semi-insulating properties with a residual concentration less than 10^{15} cm^{-3} probably due to existence of deep compensating centers. The initial experiments on deposition of the single-layer contacts with a thickness of 5 - 15 nm on the Si-doped AlGa_xN layers exhibited non-ohmic contact with high differential resistance for all metals used. The attempts to improve their performance by using different parameters of the surface pretreatment, deposition and annealing processes resulted sometimes in the ohmic contacts but with rather high TLM-resistance ($>0.5 \Omega \cdot \text{cm}^2$). Much better results were obtained for the double-layer contacts, and the lowest TLM-resistance of $8 \cdot 10^{-5} \Omega \cdot \text{cm}^2$ was achieved for the Ti/Al(15/35 nm) contact annealed at the temperature as high as 750°C for sufficiently long time (above 10 min). The influence of technological parameters (type and thicknesses of the metals, temperature and time of annealing, etc.) on contacts characteristics will be reported.

The best results on the AlGa_xN-based Schottky-type photodetectors were obtained by using Au(15nm) and Ti/Al(15/35nm) as barrier and ohmic contacts, respectively. The sequential short-wavelength shift of the photo-response curves toward deep UV (solar-blind) range with the minimum achieved cut-off wavelength less than 250 nm was observed for the structures with a Al-content as high as $x=0.7$.

[1] Y. Bilenko et al., Jap. J. Appl. Phys. **44L**, 98, (2005)

Drastic change in electronic structure of AlGaN under Ba adsorption

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III-nitride semiconductors have been the subject of intense research due to their important role for implementation of hetero- and nanostructures, optoelectronic devices operated in wide spectral range and in development of new electronic devices, e.g. HFET and HEMT where an accumulation layer plays a cardinal role.

The accumulation layer is a new nanoobject which can appear on surface of *n*-type semiconductor under condition of conduction band edge position located below the Fermi level on surface. In this case, a narrow potential well is formed. In the accumulation layer electron motion along the normal *z* to the surface is restricted, and the size quantization effect is taken place. Electron motion over the surface (*xy*) is free, and corresponding energy components are not quantized. These electron states constitute the degenerate 2D electron gas (2DEG). Accumulation layer is infrequently inherent for semiconductor surfaces. As regards III-nitrides, our studies were revealed formation of the accumulation layer at *n*-GaN surfaces under Cs, Ba adsorption [1, 2].

The Al_{0.16}Ga_{0.84}N(0001), Al_{0.42}Ga_{0.58}N(0001) samples were epitaxial layers obtained through molecular beam epitaxy with plasma activation on a sapphire substrate. Experiments were carried out at BESSY II, Helmholtz Zentrum, Berlin via synchrotron radiation by UPS with photon energies in the range of 60–400 eV. Photoemission studies were performed *in situ* in an ultrahigh vacuum at room temperature. Spectra of normal photoemission from the valence band, surface states and Ga 3*d*, Al 2*p*, Ba 4*d*, Ba 5*p* core-level were recorded.

Electronic structure of clean AlGaN surfaces and ultrathin Ba/AlGaN interfaces has been studied under step-by-step Ba submonolayer adsorption. Surface states are determined at energy of 3.2 eV, 5.4 eV, and 7.6 eV for clean Al_{0.16}Ga_{0.84}N and at energy of 3.6 eV, 5.7 eV, and 8.0 eV for clean Al_{0.42}Ga_{0.58}N. Drastic change in valence band spectra is revealed. It is found that narrowing of valence band and suppression of surface states occur with increasing Ba coverage up to 1.5 monolayer on AlGaN surfaces. At the same coverages the appearance of a new photoemission peak in the band gap close to the Fermi level are found for Ba/ Al_{0.16}Ga_{0.84}N interface. The new metallic-like photoemission peak clearly exhibits growth in intensity with increasing Ba submonolayer coverage. It is found that Ba adsorption induce cardinal change in the electronic properties with the creation of the accumulation layer. Origin of accumulation layer formation is considered.

[1] G.V. Benemanskaya et al, Surf. Sci. **603**, 2474 (2009)

[2] G.V. Benemanskaya et al, JETP Lett. **91**, 671 (2010)

Electrochemical profiling with verifying by atomic force microscopy of heterostructures with multiple quantum wells InGaN/GaN

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Methods based on space charge region alteration, such as capacitance-voltage characteristics and admittance spectroscopy, are widely used now to determine the distribution of carrier concentration and properties of energy levels (deep centers and/or quantum sized levels) in semiconductors. In [1] we have applied them for successful investigation of light emitting (LED) heterostructures with multiple quantum wells InGaN/GaN. Unfortunately, the use of mentioned methods is principally limited by reverse breakdown voltage, which imposes restrictions on achievable width of space charge region, especially for comparatively heavy doped structures.

To avoid the limitation we make use the technique of electrochemical capacitance-voltage (ECV) profiling. Here the electrolyte-semiconductor interface uses as the rectifying contact. By sequential alternation of electrochemical etching and capacitance-voltage profiling one can get gradual obtaining of charge concentration profile across *p*- and *n*-layers with the resolution as good as 1 nm practically without the limitation on depth down to the very substrate.

However, the specific feature of nitrogen growth (GaN and its solid solutions InGaN) at heteroepitaxial substrates Al₂O₃ and SiC is a great number of dislocations in the epitaxial layers (even the column-like mode of epitaxial growth). During the standard use of ECV profiling a nonuniform etching of GaN surface (InGaN) takes place, leading to systematic error in derived carrier concentration. To prevent this phenomenon the two-stage modification is more preferable: at first stage the oxidation of (In)GaN layer is employed; and at the second stage – the etching of the grown oxide. By this way the good planarity of etching surface may be obtained.

We study epitaxial LED heterostructures with multiple quantum wells (MQW) InGaN/GaN grown by MOCVD on Al₂O₃ and SiC substrates. To independently verify the etched material depth and the flatness of etching profile atomic force microscopy (AFM) is used. Basing on this data, the results obtained by classical capacitance-voltage profiling and ECV technique is comparatively discussed.

ECV measurements in "depletion profile" regime made it possible to get charge concentration profiles of InGaN/GaN MQW heterostructures at different stages of technological process with resolution, sufficient to separate a quantum well in MQW system. In "etching profile" regime the widths and concentrations in *n*- and *p*-layers InGaN, as well the position of metallurgical contact in *p-n*-junction, are obtained. Taking into account the AFM scans of ECV etching crater, a real profile of carrier concentration in depth of the samples with InGaN/GaN MQW is restored.

- [1] O.V. Kucherova, V.I. Zubkov, E.O. Tsvelev, I.N. Yakovlev, and A.V. Solomonov. *Inorganic Materials* **47**, 14, 1574 (2011)

Ultrashort laser pulses GaN ablation mechanisms

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Laser ablation under exposure to ultrashort laser pulses was shown to be promising technique for processing and machining of transparent materials such as nitride semiconductors, sapphire, and other hard and inert materials. The ablation process was investigated in using fs-laser pulses for the case of surface processing of GaN epilayers deposited on the sapphire substrate, fused silica, and other materials. Clean pits were obtained in all experiments under a single-shot irradiation. No signs of melting, no debris of re-deposition were found. There was no also collateral damage of the not-irradiated regions. The results obtained testify that the femtosecond laser ablation of transparent materials keeps the chemical properties of the ablated surface unchanged.

The nonlinear mechanism of optical power consumption has been considered. This mechanism occurs in the transparency region of the solid under irradiation with ultrashort laser pulses of high-intensity (within the multi-TW/cm² range). This irradiation is a precise technique for surface processing of semiconductors and dielectrics, particularly of hard and inert semiconductor nitrides. The typical range of treatment parameters is within the power densities of 30-50 TW/cm² at pulse width of ~135 fs and wavelength of 400 nm for the process of laser ablation in GaN. The effective absorption under this irradiation is estimated as ~2.5x10⁴ cm⁻¹, which is 20-100 times greater than the background linear absorption in GaN.

For the regime of high laser intensities the ionization time can be shorter than the pulse duration due to dependence of w on the laser intensity. Under the treatment regime with multi-TW/cm² laser pulses considered tunneling ionization dominates and total ionization occurs during the period of 10 fs or even shorter. This period is much shorter than the pulse duration.

The tunneling absorption model is used. This model takes into account quite high intensity and relatively low frequency of irradiation. The tunneling absorption mechanism has been found to be the most efficient one for wide gap semiconductors and dielectrics processed with multi-TW/cm² and femtosecond laser pulses. The laser-induced damage threshold has been determined. The correlation of the threshold with the band gap is demonstrated. The threshold increases as nearly the third power of the band gap in satisfactory agreement with the experimental results.

On the study of growth of InN by PAMBE

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The InN (0001) was grown on GaN/sapphire by Plasma Assisted Molecular Beam Epitaxy (PAMBE) with a GaN buffer layer. By controlling the growth rate ratio of In and N, the InN layers were grown in In-rich, N-rich and equivalent regions respectively. The growth of InN was monitored by reflection high energy electron diffraction (RHEED) and characterised by in-situ Scanning tunnelling microscopy (STM). The surface features were studied by ex-situ X-ray diffraction (XRD), Atomic force microscopy (AFM) and Scanning electron microscopy (SEM). A series of InN grown with different growth rates were investigated and the growth mechanism of InN layer was illustrated by the growth profile.

Effects of Crystallography-etched Facets on Patterned Sapphire Substrates for GaN-based LEDs

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High quality GaN epilayer with a low dislocation density grown on a two-step wet etching patterned sapphire substrate (TSPSS) is demonstrated by metallorganic chemical vapor deposition. The initial growth mode of epilayer was modulated through changing the second step wet etching time, which not only lead the epilayer dislocation density to be reduced 10^7 cm^{-2} level but also confine the dislocations position, corresponding to the center of the top- and bottom-side of pattern. For TSPSS-LED performance, the IQE value showed 1.2 times higher than that of conventional LEDs. At 350 mA injection current, the output power of TSPSS-LED is enhanced 41.8% as compared with conventional LEDs. These results above indicate that the TSPSS-LED has significant potential for use in future high power vertical LED applications.

Improving the internal quantum efficiency of GaN by removing the dislocations

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GaN-based LEDs are usually fabricated on lattice mismatched substrates, such as sapphire, which leads to large dislocation density in the epi-layer. Dislocations are the non-radiative centers and consequently downgrade the internal quantum efficiency (IQE). Although the blue LEDs are less sensitive to dislocations, however, they are still affected by dislocations as they are operated at high current. The short wave-length LEDs, such as near-UV LEDs or UV LEDs, are extremely sensitive to dislocations; therefore, to reduce the dislocation density is a important subject for improving the performance of GaN-based LEDs. In our previous work, we found that GaN can be decomposed in a hydrogen environment at high temperature. The surface of the GaN epi-layer etched in hydrogen is decorated by a lot of cavities, and most of the cavities are formed at the dislocation sites. In this work, we used hydrogen etch to remove the dislocations in the epi-layer. Transmission electron microscopy (TEM) and cathodoluminescence (CL) are utilized to exam the dislocations in the epi-layer; photoluminescence (PL) is utilized to exam the increasing of IQE. TEM and CL images determined that the dislocations have the priority of being etched. Consequentially, the dislocation desity were deminished after hydrogen etching. We used PL to evaluate the IQE values before and after etch, and the result shows that the IQE of the H₂ etched sample increase by 23%, which substantiated the improvement of IQE.

A-plane GaN thick films grown by hydride vapor phase epitaxy

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GaN-based optoelectronic devices grown in c-plane orientation suffer strong polarization electrostatic fields along the growth direction. The polarization fields consisting of spontaneous and piezoelectric polarization effects lead to the spatial separation of the electrons and holes within quantum wells, which results in the recombination efficiency decreasing and red-shift of emission wavelength for the devices. The phenomenon is referred to as quantum-confined stark effect (QCSE). In order to annihilate QCSE, homogeneously growing these devices on non-polar GaN substrates is most effectual method.

In this work, we used a two step growth method to grow a-plane GaN on r-plane sapphire by hydride vapor phase epitaxy (HVPE). In the first step, the growth condition was designed to induce a vertical growth trend. The surface of GaN was covered by a lot of stripes aligning along m-direction after first growth step. The morphology of the stripes observed by cross-sectional SEM images was triangular shape. Then, the growth condition was changed to enhance lateral growth trend to coalesce in the second step. The thickness of the resulted a-plane GaN thick film was approximate 100 μm . Cross-sectional SEM images showed that there were some voids in the a-plane GaN films. The production of the voids was similar to the epitaxial result using epitaxy lateral overgrowth (ELOG) method. In addition, by high-resolution X-ray diffraction (HRXRD) examination, the full width at half maximum (FWHM) was estimated to determine the crystal quality.

Single-wire photodetectors and light emitting diodes based on GaN/InGaN radial nanowire heterostructures

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Radial heterostructures in nanowires open prospects for the development of novel optoelectronic devices combining a small cross-section with an extended active region. Here, we report on the fabrication of high-sensitivity photodetectors and efficient light-emitting diodes (LEDs) based on MOVPE-grown single GaN wires containing InGaN radial quantum wells (QWs).

The n-doped GaN wires with a length of 10-20 μm and a diameter of 500-1000 nm are coated at the top part by five $\text{In}_x\text{Ga}_{1-x}\text{N}$ /GaN QWs and a p-doped GaN shell [1]. The QWs cover both the non-polar lateral side-walls and the polar upper surface. Samples with different indium content in the QWs system are studied. For the device fabrication, the wires were dispersed and contacted using electron-beam lithography.

Single-wire LEDs have been demonstrated based on *p-i-n* structures. An intense electroluminescence (EL) is observed from cryogenic to room temperature under forward bias $V > 9\text{V}$. The emission is peaked for the sample with low indium content in the blue spectral range at $E = 2.6\text{ eV}$ with a broadening of $\sim 250\text{ meV}$. For higher driving voltages, a second peak appears in the EL spectra around 3.1 eV . By using a simple model of two diodes connected in parallel, we interpret this behavior as the electrical activation of two different paths, one related to the radial heterostructure (at high energy) and a second related to the axial heterostructure (at low energy). This attribution is confirmed by the study of the nano-cathodoluminescence and on the temperature dependence of EL [2]. Similar results have been obtained for *p-i-n* structures with higher indium content.

The study of the optoelectronic properties of these devices has been completed by photocurrent spectroscopy, optical beam induced current spectroscopy and electron beam induced current measurements. Finally, we show that photodetecting structures based on *n-i-n* junctions respond to visible and UV light with energy above 2.6 eV . A responsivity as high as 8.3 e3A/W is measured for the device under 5 V bias illuminated with 360 nm light [3].

[1] Koester et al., *Nanotechnology* **21**, 015602 (2010)

[2] Jacopin et al., *Applied Physics Express* **5**, 014101 (2012)

[3] De Luna Bugallo et al., *Applied Physics Letters* **98**, 233107 (2011)

Optical properties of the InGaN light-emitting diodes grown on different-shaped pattern sapphire substrates

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The InGaN-based light-emitting diode (LED) structures were grown on the flat-form (FPSS) and the pyramid (PPSS) pattern sapphire substrate through through a metalorganic chemical vapor deposition system. Both of the pattern sapphire substrates were fabricated through the wet etching process in a hot H₃PO₄ solution. In the photoluminescence measurement, the internal quantum efficiency (IQE) was measured at 76.1% and 83.8% for the FPSS-LED and the PPSS-LED, respectively. At 10K, the PL peak wavelength blue-shifted of FPSS-LED and PPSS-LED were measured as 2.0nm and 0.7nm by varying the excitation laser power. In the reverse-bias PL spectra, the flat-band voltages were measured at -12V and -8V for FPSS and PPSS structures, respectively, that indicated the piezoelectric fields in the PPSS-LED had been reduced. The light output power of PPSS-LED was higher than the FPSS-LED structure by increasing the IQE values and reducing the piezoelectric field in the InGaN active layer grown on the pyramid (PPSS) pattern sapphire substrate.

AUTHOR INDEX

A		H.E. Beere	84
M. Adachi	220, 264	A. Behrends	100, 203
R. Aidam	213, 214	S. Bellei	206
I. Akasaki	91, 210	E. Bellet-Amalric	61
R.Kh. Akchurin	197, 245	K. Bellmann	258
H. Akiyama	59	E.Yu. Belousova	184
M. Albrecht	160	A.E. Belyaev	176, 177, 278
D. Alexandrov	113, 221, 222	K.G. Belyaev	165
I. Alexandrov	92	T. Ben	214
A.N. Alexeev	257	G. Benemanskaya	280
M. Ali	97	E. Beraudo	125, 204
N. Alkeev	144	J. P. Bergman	110
B. Alloing	204	K.A. Bertness	206
D.W.E. Allsopp	125, 159	F. Bertram	50
E. Alves	164	V.N. Bessolov	192, 215
O. Ambacher	213, 214	R. Bhat	72
H. Amano	216	T.N Bhat	270, 271
L. M. Amorim	111	A. G. Bhuiyan	107
B. Amstatt	199	P. Binsted	221, 222
A. Antipov	121, 154, 193	J. Birch	194
Y. Aoyagi	122	A. Birukov	225
T. Araki	70	E. Blanquet	227, 228
A. Arendarenko	65	N.I. Bochkareva	135
I. Argut	50	M. Boćkowski	73
O. Ariyada	198	R. Boichot	227, 228
E. Armour	96	A.D. Bolshakov	80
A. Arnatkevičiūtė	277	N.S. Boltovets	278
L. Artús	101	A. Bonanni	82
D.M. Artemiev	97	R. T. Bondokov	52
D.J. As	67, 145, 175	I. Booker	138
P. Aseev	223, 251	C. Bougerol	79, 105, 199
S. Averin	144	V.E. Bougrov	97
N. Averkiev	149	C. R. Bowen	159
E.A. Avramenko	177	A. Boyd	98
A. Avramescu	71	J. Brault	68, 104
V. Avrutin	180	H. Bremers	209
M. Azize	137	M. Brendel	55
B		D. Brien	231
A. Bakin	100, 203	C. Brimont	104
M. Balaji	228	V.N. Brudnyi	224
R.G. Banal	163	G. Bruederl	71
Q. Bao	132, 136	P.N. Brunkov	260
P.G. Baranov	89	F. Brunner	55
I. Barash	121, 154, 193	Y. Bu	263
K. Baskar	228	K.A. Bulashevich	139
A.A. Baski	180	M. Bürger	175
Y. Basma	164	K. S. A. Butcher	113, 221, 222
A.L. Bavencove	199	R. Butté	153

E.R. Buß	209	J.G. Correia	111
Yu. Buzynin	225	N. Coudurier	227, 228
C		P. M. Coulon	204
A. Cabaj	200	S. Craft	51
H. Çakmak	226	A. Crisci	228
A. Cantarero	78	A. Cros	102
J.-F. Carlin	153, 158	R. Cuscó	101
D. Carvalho	214	R. Czernecki	73, 238
A. Castiglia	153	D	
F. Causa	125	J.-J. Dai	288
L. B. Cen	62	R. Dalmau	51
V.P. Chaly	140, 257	B. Damilano	63, 104, 109, 126
H. Chang	276	R. Dargis	108
Y. H. Chang	167	A. Das	61
J.-M. Chauveau	68	B. Daudin	102, 105
C.-L. Chen	286	M. Dauelsberg	231
G. Chen	69	A.V. Davydov	206
J. Chen	52	V.Yu. Davydov	92, 168, 186, 212, 260
P. P.-T.Chen	113, 138	P. Dawson	94
W. Chen	242	A. De Luna	287
X Chen	98	Bugallo	
X. J. Chen	79	S. Decoster	111
Y.-T. Chen	194	E.A. DeCuir Jr.	176
Z.Z. Chen	155, 156, 157	F. Demangeot	102, 126
S. Chenot	63, 126	P. De Mierry	104
N. Cherkashin	95	P. Demirel	226
A.E. Chernyakov	139, 149, 150	D. Detert	85
V. Chernyshov	143	T. Dietl	83
S.F. Chichibu	99, 132, 136, 170	M. Dineen	125
M.W. Cho	218, 219	L. Dobos	92
Y.-H. Cho	166, 167	B. Doisneau	228
J. H. Choi	244, 262	I.P. Dolbnya	93
S. Choi	119, 141	N. Domènech-Amador	101
Y.J. Choi	235	F. Donatini	269
J. Christen	50, 87	R. Doradziński	56
C.-M. Chu	285, 286	J. E. Downes	113
M. Chubarov	208	P. Drechsel	160
A. Clark	108	M. Drozdov	225
A. Claudel	227, 228	Yu. Drozdov	225
P.H. Clifton	90	O. D. Dubon	85, 112
S. Coindeau	227, 228	J.-Y. Duboz	109
R. Collazo	51	V.G. Dubrovskii	80
M. Conroy	261	A. Dudin	140
V. Consonni	80	A.I. Duff	229
M. Cooke	125	R.D. Dupuis	119, 141
Y. Cordier	63, 109, 126	C. Durand	79, 199, 201, 287
P. Corredera	183	M. Durnev	169
		A. Dussaigne	199

R. Dwiliński	56, 181	S. Gerhard	71
	E	S. R. Gibb	52
M. Edwards	159	S. Gieraltowska	200
P.R. Edwards	93	P. Gilet	199
C. Eichler	71	N. Gladisheva	144
S. Einfeldt	55, 74	G. Gommé	109
P.G. Eliseev	282	V.V. Gomonovych	176
A. Emen	226	R. Gong	141
B. M. Epelbaum	128	M. González-Herráez	183, 248, 249
F. Erdem Arkun	108	F. González-Posada	77
S. Erenburg	92	N. Gonzalez Szwacki	83
M. Eriksson	273	R.I. Gorbunov	135
I. Ermoshin	65	T. Goto	134
J. Eymery	79, 199, 201, 287	H. Gotoh	134
	F	A.V. Govorkov	207
R. Félix	214	N. Grandjean	48, 153, 158
V. Fellmann	227, 228	J. R. Grandusky	52
O. Feron	98	A. Greshnov	150
P. Ferret	199	V. Grillo	160
A. Fischer	141	T. Grinys	205, 236, 277
U. Forsberg	138, 252, 273, 274	I. Grzegory	73
M.A. Foussekis	180	T. Guillet	104
C. T. Foxon	85, 112, 253	A. Gurary	96
N. Franco	164	A.S. Gurin	89
E. Frayssinet	109, 126		H
D. Frolov	281	J.-S. Ha	218, 219
X.X. Fu	156, 157	S. Hagedorn	230, 247
S. Fujikawa	54	T. Hager	71
T. Fujimori	124	J. Han	106, 166
H.Fujioka	49, 75, 195, 239, 254, 256, 265	M. Han	117, 234
T. Fujishima	70	N. Han	117, 234
K. Fujito	170	T. Hanada	244, 262
Y. Fukuhara	103	K. Hanaoka	232
H. Fukuyama	129, 220, 241, 264, 265	A. Hangleiter	209
M. Funato	163	H. Harima	171
	G	A. Hashimoto	107, 171
B. Gaddy	51	M. Hayashi	233
R. García	213, 214	K. Hazu	170
N. Garro	78	H. Helava	57, 121, 154, 193
B. Gayral	79, 105	C. Hemmingsson	123, 190
P. Gečys	236	A. Henry	208
L. Geelhaar	80	M. den Hertog	269
V. Georgiev	113, 221, 222	K. Hestroffer	105
D. Georgieva	113, 221, 222	M Heuken	98, 173, 231
R. Gergova	221, 222	M. Higashiwaki	86

M. Himmerlich	214	S. Ishizawa	76
C. Himwas	269	I.G. Ivanov	138
K. Hiramatsu	246	S.V. Ivanov	165, 182, 223, 251, 279, 280
T. Hirasaki	232	T. Iwabuchi	217
H. Hirayama	54	M. Iwaya	91, 210
D. Hironaga	107	I. Izmaylova	57
H. Hofer	203		J
V. Hoffmann	74	G. Jacopin	206, 287
H. Högberg	208	J. Jang	116, 276
P.O. Holtz	273, 274	L.-W. Jang	81, 207
T. Honda	86, 233	E. Janzén	138, 252, 273, 274
C.-H. Hong	117, 234, 275	K. Jarašiūnas	178, 277
S.-K. Hong	218, 219	E. Jelmakas	236
M. Honjo	124	D.-W. Jeon	81, 207
R.-H. Horng	284	H. Jeon	276
T. Hossain	126	M.-H. Ji	141
T. Hotta	114	S.Y. Ji	262
C.-L. Hsiao	194	Q. Jiang	159
C.-H. Hsieh	288	S. Jiang	157
C.W. Hsu	273, 274	X.Z. Jiang	156, 157
Y.-C. Hsu	285	R. J. Jiménez	101
H.-H. Hsueh	284	Riobóo	
X. Hu	155, 242	V.N. Jmerik	66, 165, 182, 223, 251, 279, 280
Z. Hu	157	T. Jones	283
C. C. Huang	62	K. Joo	116
T. Huang	64	M. Jublot	204
M. Hugues	125, 204	F.H. Julien	175, 206, 287
L. Hultman	194, 273		K
C.J. Humphreys	84, 94	V. Kachkanov	93
L. Hussey	51	A. Kadir	258
J. Hwang	235	A. Kadri	142
S.-J. Hwang	272	A. Kadys	178, 205, 236, 277
M. Hytch	95	A. Kahouli	104
		A. Kakanakova-Georgieva	252
	I	K. Kakimoto	128
T. Igaki	86	M. Kakuda	103
D. Iida	91, 210	A.T. Kalghatgi	270, 271
I.V. Ilyin	89	H. Kalisch	173
H. Imabayashi	58, 124, 127	A.E. Kalmykov	215
M. Imade	58, 124, 127, 263	A. Kalpakovaitė	205
M. Imanishi	127	S. Kamiyama	91, 210
T. Inamoto	255	J.H. Kang	117, 234, 275
S. Inoue	75, 195, 239, 265	X. Kang	266
D. Irving	51	Y. Kangawa	128
M. Isemura	263	M.J. Kappers	84, 94
T. Ishiguro	132, 136	G. Karavaev	143
Y. Ishikawa	170	K.F. Karlsson	273

S.Yu. Karpov	139, 169	N.G. Kolin	224
Y. Kashima	54	A.F. Kolomys	177
R. Katayama	103, 217, 244, 262	T. Komissarova	279
Y.S. Katharria	234	R.V. Konakova	177, 278
M. Kato	129	T. Kondo	195
M. Katoh	75	Y. Kondo	91, 210
N. Katsavets	140	E.V. Konenkova	192
N.A.K. Kaufmann	158	Kosobutsky	224
Y. Kawakami	163	Y. Kotsar	61
O. Kazarova	57	A.R. Kovsh	97
B. Kemp	221	A. Koukitu	114, 232
P. Kempisty	152, 237, 238	Yu.V. Kozhanova	174
S Khromov	110, 190	N.A. Kozlovskaya	282
O. Khrykin	225	D.M. Krasovitsky	140, 257
D.-M. Kim	272	N. Kriouche	104
D.-U. Kim	276	O.N. Krokhin	282
H.J. Kim	119	S. Krukowski	152, 237, 238, 267
H.K. Kim	117, 234	S.B. Krupanidhi	270, 271
H.-S. Kim	235	F. Krzyzewski	240, 267
H.Y. Kim	117, 234	T. Krzyzewski	283
J. Kim	141	S. Kuboya	103, 255
J.-H. Kim	167	R. Kucharski	56, 181
J.-H. Kim	235	O. Kucherova	281
J.-K. Kim	272	S. Kück	203
Y.-H. Kim	167	V. Küller	55
T. Kimura	217	R. Kudrawiec	181
L. Kirste	213, 214	Ya.Ya. Kudryk	278
E. Kishikawa	239, 265	V. Kueller	120
K. Kishino	76	S.A. Kukushkin	192, 215
Yu.E. Kitaev	168	T. Kumada	241
A. Kitamoto	58, 124, 263	Y. Kumagai	114, 232
P. Kivisaari	147	M. Kumar	270, 271
V.P. Klad'ko	278	P. Kumar	196
M. Klein	130	S. Kumar	244
K. Klosek	200	V.S. Kumar	262
A. Knauer	120	V.A. Kureshov	197, 245
M. Kneissl	53, 74, 120, 258	S. Kurin	121, 154, 193
A. Knigge	55	N. Kurose	122
S.M. Knoll	84	M. Kurouchi	122
A. Knübel	214	N. Kuwano	128
K. B. Ko	117, 234	P. Kuznetsov	144
S.-M. Ko	167	Ya.V. Kuznetsova	165, 172, 251
Y.-H. Ko	166, 167	R. Kyutt	92, 168, 186, 211 , 212
A. Kobayashi	195, 254, 256		L
M. Kociak	105, 287	Y. Lacroix	131
K. Kodama	171	I. Lamkin	154, 279
Y. Koide	148	S. Lapinskas	277
S. Kokin	140	M. Lapushkin	280
T. Kolbe	53	D.J. Larson	90

P.E. Latyshev	135	X. Liu	266
K. M. Lau	64	Y. Liu	217
P. Lauffer	98	Y.H. Liu	262
P. Lavenus	206, 287	Z. Lochner	119
J.H. Leach	180	K. Lorenz	93, 164
M. L. Lebby	108	M. Lozac'h	202
J. LeBeau	51	S. Luca	228
C. Leclere	105	M Luenenbuerger	98, 231
C.-H. Lee	285, 286	A. Lukyanov	225
H.-J. Lee	218, 219	W.V. Lundin	95, 161, 168, 212, 243, 259, 260
H.-Y. Lee	235	E.Yu. Lundina	260
J.-M. Lee	272	A. Lundskog	138, 273, 274
J.S. Lee	235	W. Luo	266
J. Y. Lee	167	E.V. Lutsenko	173, 178
I.-H. Lee	81, 207	L. Lymperakis	160, 229
Y.-C. Lee	119	V. Lysak	275
S.-N. Lee	272		M
S.W. Lee	218, 219	Z. Mahfoud	105
W.-I. Lee	285, 286	J. A. Majewski	83
R Leiers	98	Yu. Makarov	57, 121, 154, 193
Y.S. Lelikov	135	T. Malin	92, 185, 186
H. Lengner	50	T. Malinauskas	178, 205, 236, 277
T. Lermer	71	J. Malindretos	78
M. Leroux	68, 104, 109, 126	V.V. Mamaev	257
Le Si Dang	269	M. Mandl	125
M. Leszczyński	73, 238	J. M. Mánuel	213, 214
B. Leung	106, 166	T. Markurt	160
R. Leute	50	A.A. Marmalyuk	197, 245
A. Levander	85, 112	H. Mariette	201
M. Levinshtein	149, 150	D. Martin	158
M. Leyer	258	R. Martin	227, 228
D. Li	155, 242	R. W. Martin	85, 93
H. Li	268	M. Maruyama	58, 124, 127
H.N. Li	261	T. Maruyama	250
J.Z. Li	156, 157	J. Massies	104, 109
L. Li	242	K. Masumoto	124, 127
X. Li	180	E. Matioli	70
M. Liao	148	H. Matsubara	91, 210
Z. Liliental-Weber	112	H. Matsumura	217
T.-Y. Lim	235	D. Matsuo	58, 124, 127
C.-F. Lin	288	T. Matsuoka	103, 217, 244, 262
C.H. Lin	250	C. Mauder	173
C.-M. Lin	288	A.V. Mazalov	197, 245
J Lindner	98	Yu.I. Mazur	176
D. Litvin	57, 193	J.D. McNamara	180
L. Liu	155, 242	F. Mehnke	53, 258
N. Liu	155	T. Meisch	50
P. Liu	242	C. Meissner	258
S.T. Liu	69	A. Mekys	205

T. Menkad	222	J. Neugebauer	160, 229
E. Menkovich	154, 279	A.E. Nikolaev	95, 161, 188, 243
A. Merkulov	187	V. Nikolaev	188, 192
S. Metzner	50	A.S. Nikolenko	176
M.M. Mezdrogina	174	D. Nilsson	252
C. Mietze	175	H. Nishihara	54
A. Mihara	107	T. Nomura	246
S.M.C. Miranda	164	S. V. Novikov	85, 112, 253
S. Mita	51	N.N. Novikova	185
B. Mitrovic	96	H. Nykänen	189
H. Miyake	246	O	
A.M. Mizerov	165, 182, 223, 251	M.A. Odnoblyudov	97
A. Mogilatenko	74, 247	K.P. O'Donnell	93
M. Mohajerani	203	H.-K. Oh	235
E.N. Mokhov	57, 89	T. Ohachi	198
B. Monemar	110, 123, 190	T. Ohkawa	54
E. Monroy	61, 77, 183, 248, 249, 269	J. Ohta	75, 195, 239, 254, 256, 265
L. Monteagudo-Lerma	183, 248, 249	M. Ohtsuka	129, 241
B. Moody	51	J. Oksanen	147
D. Moon	116, 276	K. Okubo	254
F. M. Morales	213, 214	K. Okuno	216
M.A. Moram	84	D. Olson	90
Y. Mori	58, 124, 127, 263	K. Onabe	103, 255
K. Morita	75	C. Oppo	78
H. Morkoç	180	V. Oreshkin	65
E.S. Moskalenko	174	M. Oseki	256
A. Mukhtarova	201	M. Oshima	239, 254, 256, 265
H. Murakami	114, 232	T. Oshio	216
K. Murakami	58, 124, 127	A.V. Osipov	192, 215
Y. Muramatsu	107	E. Özbay	226
A.V. Myasoedov	215	P	
K. Mynbaev	188	A.A. Padalitsa	197, 245
M. Mynbaeva	188	T. Palacios	70, 137
N		J. Palisaitis	194, 273
S. Nagao	170	V.N. Panteleev	278
S. Nagalyuk	57, 193	A. Paranjpe	96
S. Nagata	59	P.J. Parbrook	261, 268
Y. Nakano	202	J. Park	116
H. Namita	170	M.-J. Park	272
Y. Nanishi	70, 101, 116, 276	S.H. Park	116, 276
F. B. Naranjo	183, 248, 249	Y.J. Park	117, 234
S. Nargelas	178, 277	P. P. Paskov	110
S. Naritsuka	250	T. Paskova	60, 180
D. Nechaev	182, 223, 251	B. Paszkiewicz	146, 162
M. Nemoz	68	R. Paszkiewicz	146, 162
C. Netzel	74, 133	L. Pauli	98, 231

V.N. Pavlovskii	173, 178	B. Reuters	173
R. Péchou	102	T. Rice	51
B. Pecz	92, 159	E. Richter	133, 230, 247
H. Pedersen	208	H. Riechert	80
L. M. C. Pereira	111	L. Rigutti	206, 287
S. Pereira	93	D.A. Ritchie	84
G. Perillat-Merceroz	79	L. Riuttanen	147
P. Perlin	73	A. Rizzi	78
P. O. Å. Persson	194, 273	P. Rode	125
S. Peters	203	A. Roenkov	121, 154, 193
S.I. Petrov	257	I. Roqan	164
P. Petrov	149	E.M. Roginskii	168
D. Piedra	137	M.M. Rozhavskaia	168, 212, 243, 259, 260
D. Pique	227, 228	A.E. Romanov	97
A.Y. Polyakov	81, 207	N.G. Romanov	89
F.A. Ponce	141	A.S. Romanyuk	177
A. Ponchet	102, 126	U. Rossow	209
M. Pons	227, 228	M.-A. Rothe	53
M. Pophristic	96	E. Rotunno	160
G. Pozina	110, 123, 190	R. Roucka	108
K. Prasertsuk	217	B. Roul	270, 271
E. Preble	180	M. Rudziński	181, 226
C. Prieto	101	B.D. Ryu	117, 234
M. Pristovsek	53, 258	J. H. Ryu	234
T.J. Prosa	90	J.-H. Ryou	119, 141
D.Yu. Protasov	186	M. V. Rzhetski	173, 178
	Q		S
K. Qiao	132, 136	D.R. Sabitov	197, 245
Z. Quan	261, 268	T.C. Sadler	261, 268
	R	T. Saito	70
G. Račiukaitis	236	J. Sakaguchi	70
M.K. Rajpalke	270, 271	A.V. Sakharov	95, 161, 243
M.V. Rakhlin	165	S. Sakr	175
V. Ramesh	76	G.J. Salamo	176
S. P. Rao	52	D. Salomon	79, 199, 287
L. Rapenne	183, 269	D. Sam Giao	79, 105, 199
M. J. Rashid	109, 126	L. Sang	148
V. Ratnikov	212, 251	W. L. Sarney	85
A. Razzhuvalov	143	M. Sarzyński	73
Y.T. Rebane	135	L. W. Sang	202
C. Reich	53, 120	E. Sarigiannidou	183, 269
R. dos Reis	112	T. Sasaki	124
I. Reklaitis	178	A. Sato	134
T. Remmele	160	T. Sato	134
H. Renevier	105	Y. Sato	198
S. Rennesson	63	Md.M. Satter	141
M. A. Reshchikov	100, 179, 180, 203	A.V. Sachenko	278
A. Reszka	200		

M. Scheglov	211, 212	M. Sopenan	147, 189
B Schineller	98, 231	L.M. Sorokin	215
R. Schlessner	51	M. Sowa	91, 210
W. Schmunk	203	D.M. Spiridonov	184
F. Scholz	50, 130	A.V. Spirina	186
L. J. Schowalter	52	P. Stauss	160
F. Schulte	98, 231	J. Stellmach	53, 258
T. Schulz	160	P. Strak	152, 237, 238
S. Schwaiger	50	M. Strassburg	125
T. Schwaner	258	G. Strasser	183
N. Sebkhi	141	U. Strauss	71
E. Secco	78	V.V. Strelchuk	176, 177
F. Semond	109, 125, 126	W. Strupiński	181, 226
S. Sergent	109	K. Sugita	107
E. Shabunina	149, 150	Y. Sugiura	233
Sh. Sharofidinov	192	M. Sugiyama	220, 264
V. Shashkin	225	T. Sugiyama	91, 210
M.P. Shcheglov	192	E.-K. Suh	234
B. Shen	62, 69	S. Suihkonen	147, 189
K.-C. Shen	284	T. Sumi	263
S.-C. Shen	119	M. Sumiya	148, 202
V.N. Sheremet	278	T. Suski	73
E. Shevchenko	182	S. P. Svensson	85
N. Shibata	216	Yu. Sveshnikov	65
P.A. Shields	125, 159	T	
N. Shigekawa	107	H. Takagi	54
H. Shindo	262	M. Takasugi	264
S. M. Shivaprasad	118, 196	H. Takazawa	58, 124, 127
N. Shmidt	149, 150, 191	S. Takenaka	114
K. Shojiki	244, 262	T. Takeuchi	91, 122, 210
Y.G. Shreter	135	I. Takezawa	233
S. Shvarkov	203	J. Takino	263
M. Šilinskas	205	A. Tanaka	220, 264
D. J. Silva	111	S. Tanaka	216
T. Simoneit	258	Y. Taniyasu	153
Z. Sitar	51	Y.B. Tao	156, 157
A. Sitnikova	182, 188, 251	S. Tarasov	154, 279
D. Sizov	72	M. Tashiro	54, 170
E. Skoroxodov	225	M. Tchernycheva	175, 206, 287
A. Smirnov	92, 168, 186, 212	G.	200
M.B. Smirnov	168	Tchutchulashvili	
N.B. Smirnov	207	M. Teisseire	68, 204
M. Sobanska	200	K. Temst	111
V.I. Solomonov	186	P. Terziyska	221, 222
V.A. Soltamov	89	J. Thalmair	50
O.A. Soltanovich	151	Q.T. Thieu	255
N.T. Son	252	S Thomas	98
J. Song	106, 166	K. Thonke	50
R. Songmuang	269	E.J. Thrush	94
		S. Timoshnev	280

I. Tischer	50	M.E. Ware	176
M. Tlaczala	146, 162	N. Watanabe	107
Y. Todoroki	58, 124, 127	I.M. Watson	93
D.O. Tolmachev	89	A.D. Wieck	203
R. Tomašiūnas	205, 236, 277	I.A. Weinstein	184
A.A. Toropov	165, 182	T. Wernicke	53
L. Tóth	159	M. Weyers	55, 74, 120, 133, 230, 247
O. Tottereau	204	A. Wierzbicka	200
G. Tourbot	105	A. Wolfson	57
A. Trampert	80	M. Wosko	146, 162
G. Tränkle	74, 133, 247	J. Wu	85, 112
X.T. Trinh	252	J. Wu	266
S.I. Troshkov	243, 259, 260	Y.-H. Wu	285, 286
S. Trubina	92	D.-S. Wu	284
A.F. Tsatsulnikov	95, 161, 243, 259, 260		X
D. Tsvetkov	206	Y. Xia	68
I. Tsyplenkov	65	J. Xie	51
A.I. Tsyuk	135	Y. Xie	242
F. Tuomisto	189	F. J. Xu	62
J. Tweedy	51	J. Xu	116, 276
	U	Z. Y. Xu	62
S. Uchiyama	250		Y
K. Udvary	180	G.P. Yablonskii	173
A. Uedono	99, 170	M.A. Yagovkina	168, 212, 243, 260
N. Uematsu	70	V.A. Yakovlev	185
K. Ueno	239, 265	E.B. Yakimov	151, 191, 207
A. Urban	78	T. Yamaguchi	70, 86, 101, 233
S.O. Usov	161	A. Yamamoto	107
	V	Y. Yamamoto	198
S. Valdueza-Felip	61, 183, 248, 249	K. Yamano	76
P. Valvin	104	C.-C. Yang	288
A. Vantomme	111	R. Yang	283
A. Vasiliev	57, 193	W. Yang	155
P. Vennéguès	63, 68, 104, 204	Z. Yang	242
P.S. Vergeles	191	Z. J. Yang	62
A. Vescan	173	T. Yao	134, 218, 219
K. Vinogradova	188	B.S. Yavich	177
A.S. Vokhmintsev	184	Y.-H. Yeh	285, 286
V.V. Voronenkov	135	C. Yerino	166
	W	P.D. Yoder	119, 141
A. Waag	100, 115, 203	C. Yokoyama	132, 136
M. Wada	198	J. M. Yoon	167
U. Wahl	111	E. Yoon	116, 276
W. Walukiewicz	85, 112	S. Yoshikado	198
J. Wang	102	M. Yoshimura	58, 124, 127, 263
S.Y. Wang	156	D.-J. You	116
X.Q. Wang	62, 69	S. W. Youn	54
W. N. Wang	159	K.M. Yu	85, 112

T. Yu	266
T.J. Yu	156, 157
T.-Y. Yu	285
M. Yutani	70
Z	
A. Zado	145
L.F. Zagonel	287
C. Zah	72
M. Zając	56
A.L. Zakgeim	139,150
L. Zakharov	144
M. Zaluska-Kotur	240, 267
M.V.	165, 172
Zamoryankaya	
E.E. Zavarin	161, 168, 243, 259, 260
I.N.	282
Zavestovskaya	
U. Zeimer	74, 120, 133, 230, 247
G.Y. Zhang	156, 157
G. Zhang	242
G. Zhang	266
Y. Zhang	106, 119
S. Zhang	84
Y. Zhang	217
Yu.V. Zhilyaev	278
V. Zhitov	144
D. Zhu	94
X. Zhu	64
K. Zhuravlev	92, 185,186
K. Zitouni	142
V. Zubialevich	261, 268
V. Zubkov	281
A. Zubkova	281
A.S. Zubrilov	135
A. Žukauskas	178
J. Zúñiga-Pérez	125, 204
J. Zweck	50
Z.R. Zytikiewicz	200



FUND FOR INFRASTRUCTURE AND EDUCATIONAL PROGRAMS

The Fund for Infrastructure and Educational Programs was founded during the reorganization of the Russian Corporation of Nanotechnologies. It stimulates nanotechnology infrastructure building to support innovation in Russia. The Fund for Infrastructure and Educational Programs primarily focuses its activity in these areas:

- Formation of infrastructure for nanotechnology
- Development of human resources for nanoindustry
- Market development for nanotechnology products
- Improvement of the legislative framework for innovation
- Standardization and certification of nanoproducts and evaluation of their safety
- Refinements in metrology
- Popularization of nanotechnology and nano-enabled products

The highest governing body of the fund is its Supervisory Council, which is chaired by Alexey Ponomarev, Vice President, Skolkovo Institute of Science and Technology (Skolkovo Tech). In accordance with the charter of the fund, the Council determines priorities for its activities, establishes its strategies, and sets its budget.

The Management Committee is the fund's collegial executive body. It is chaired by RUSNANO CEO Anatoly Chubais. Andrey Svinarenko is CEO of the Fund for Infrastructure and Educational Programs.



Technology infrastructure: Nanotechnology centers

Nanotechnology centers (nanocenters) are an indispensable element of infrastructure for developing Russia's nanoindustry. They incubate start-ups and prepare small innovative companies for market entry.

The day-to-day functions of the nanocenters, which numbered 12 at the end of March 2012, include:

- Engineering development for nanotech products, experimental design and experimental technological work for commercial customers
- Intellectual property protection
- Management, patent, and marketing support to small innovative companies
- Technology licensing
- Testing and obtaining product certifications

Nanocenters are selected through tenders organized by the Fund for Infrastructure and Educational Programs. By the end of the first quarter of 2012, twelve projects had been selected. Their budgets total \$930 million of which RUSNANO will finance \$450 million.



Technology infrastructure: Engineering companies

Engineering companies develop technology and instruments and product prototypes on contract basis, using their own facilities and equipment to devise solutions for specific customers.

Engineering companies differ from manufacturing projects whose goals are to produce and sell products in the mass market. The work of an engineering company is usually unique for each customer and does not go into replication.

In the competitions that had been conducted through the first quarter of 2012, three companies were awarded rights to enter into investment agreements for engineering projects with the Fund for Infrastructure and Educational Programs.



Educational Programs

For successful development, Russia's nanoindustry will need 100,000 highly qualified specialists, each thoroughly knowledgeable in his or her field and possessing a multidisciplinary perspective. The Fund for Infrastructure and Educational Programs has approached this challenge through requests to the country's education system, which has provided the base for educational activity – programs in skill development and advanced education, foremost for employees of RUSNANO's portfolio companies. Through March 2012 the fund had sponsored 63 programs in which 1,100 individuals have studied.

The fund's educational programs fall into two groups: programs with an engineering and technology focus and programs that train managers from the nanoindustry in leadership and management skills. Most educational programs for portfolio company staff incorporate international collaboration: study in foreign universities, onsite learning in businesses in Europe and the United States, lectures by world's leading specialists.

The educational programs of the fund draw on distinguished Russian scientific and educational centers and foreign universities and companies, among them: Stockholm School of Economics Russia, MIT and Babson College (USA), Feng Chia University (Taiwan), Technical University of Berlin (Germany), Kember Associates and Strategic Technologies Practice (United Kingdom), Alcatel-Lucent (Ireland), Bell Labs and Skylight Navigation Technology (USA), OMMIC (France), and Optogan's Dortmund research center (Germany).

Concerned that future generations should find work as nanoindustry professionals, the fund has initiated programs for early professional orientation of students and methodological support for teachers. The work is the province of the fund's RUSNANO School League, which has already been extended to 20 schools in 10 regions of the Russian Federation. The annual all-Russia olympiad jointly conducted with Moscow State University, Nanotechnology – Breakthrough to the Future! plays a high-profile role in educating young people to look toward the nanosciences.



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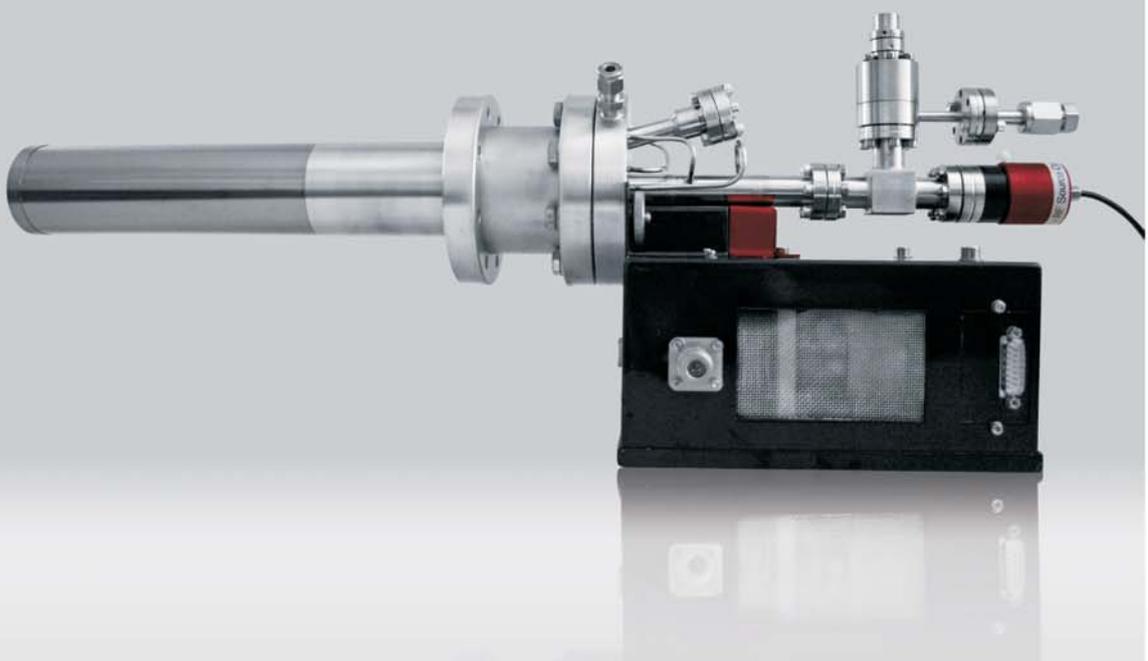
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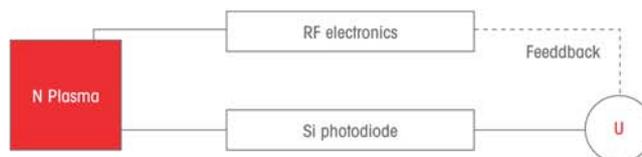
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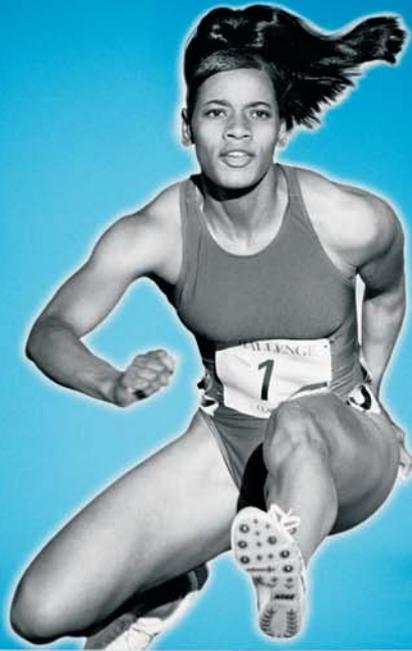


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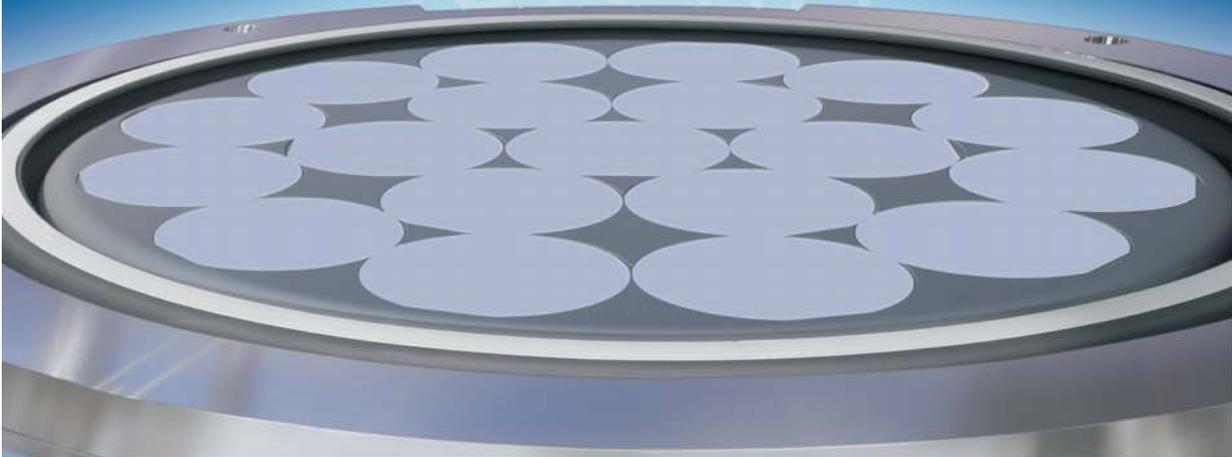
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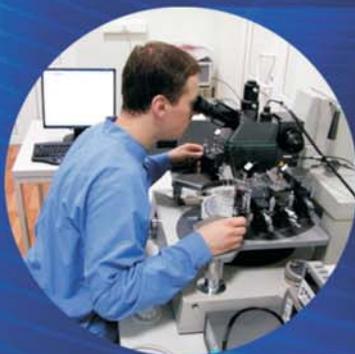
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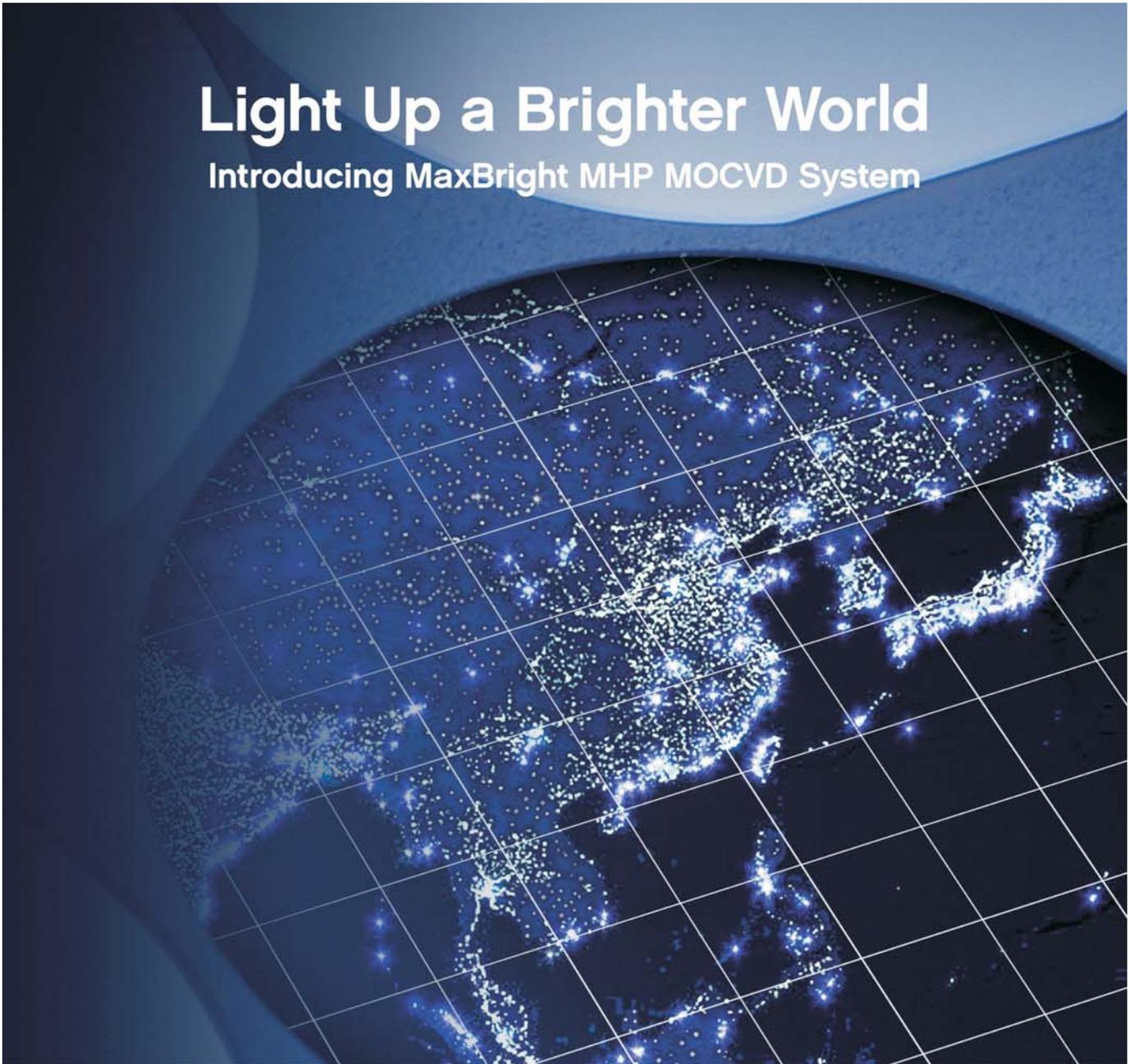
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Optogan is a vertically integrated Russian LED & luminaire manufacturer. The company is one of the technology leaders in developing and producing high brightness light-emitting diodes (HB LEDs) for a large variety of applications including Solid State Lighting. Full vertical integration from crystal growth to luminaires on a global level enables competitive, innovative and cost-efficient LED solutions.

Optogan Oy was established in 2004 in Helsinki by Maxim Odnoblyudov, Vladislav Bougrov, and Alexey Kovsh – graduates of the Ioffe Physical-Technical Institute and students of Nobel Laureate Zhores Alferov. The company's



basic technological operations are carried out in the German city of Dortmund, where a nanodiode production line using Optogan's unique technology was based. In 2009, CJSC Optogan was registered in Russia in order to establish a Russian manufacturer of high-brightness LEDs. Rosnano, the Onexim Group, and RIK stepped forward as investors in the project.

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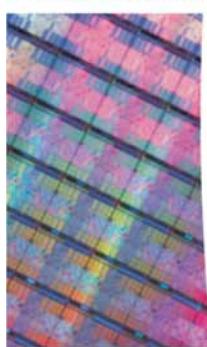
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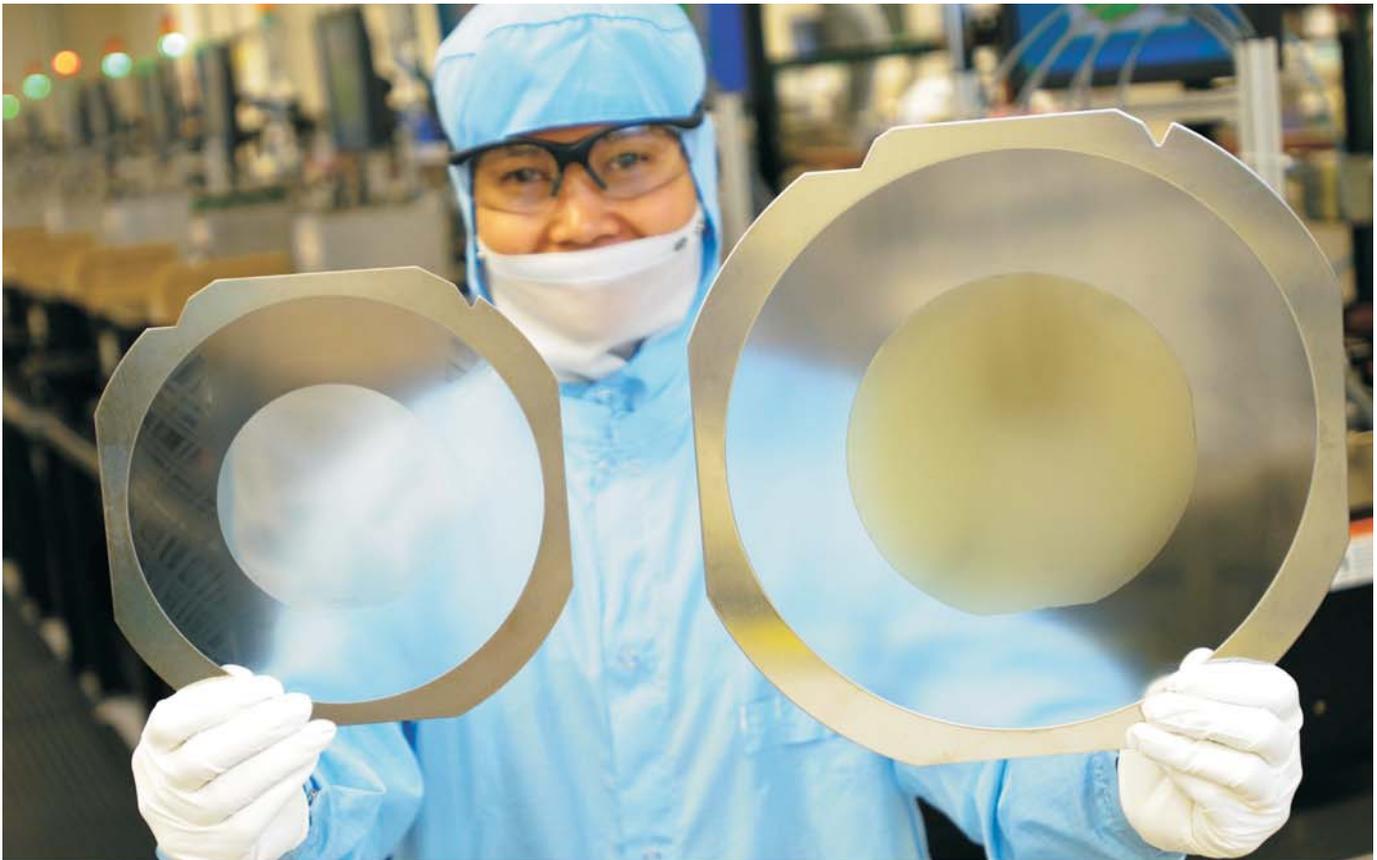
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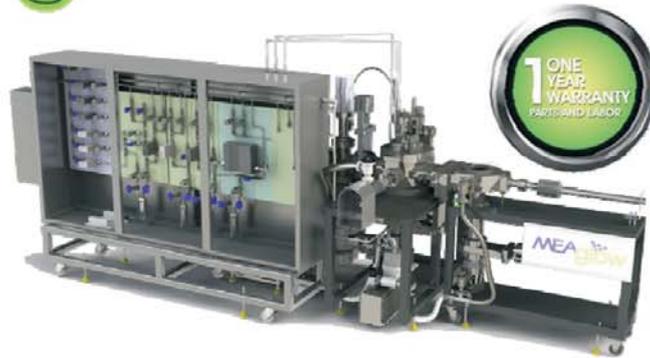
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