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Amorphous grain boundary layers in the ferromagnetic nanograined ZnO films

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ABSTRACT

Pure ZnO thin films were obtained by the wet chemistry ("liquid ceramics") method from the butanoate precursors. Films consist of dense equiaxial nanograins and reveal ferromagnetic behaviour. The structure of the ZnO films was studied by the high-resolution transmission electron microscopy. The intergranular regions in the nanograined ZnO films obtained by the "liquid ceramics" method are amorphous. It looks like fine areas of the second amorphous phase which wets (covers) some of the ZnO/ZnO grain boundaries. Most probably these amorphous intergranular regions contain the defects which are responsible for the ferromagnetic behaviour.

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1. Introduction

Using the so-called p-d Zener model Th. Dietl et al. have theoretically predicted that ZnO and other oxides doped by "magnetic" atoms like Co, Mn or Fe can possess ferromagnetic (FM) behavior with a high Curie temperature T_c above room temperature (RT) [1]. This is due to carrier-related FM interactions, where the FM ordering of the transition metal (TM) ions is induced by a magnetically polarized and doping modified ZnO host. The paper [1] followed by a huge amount of experimental and theoretic work devoted to the search or predicted RT ferromagnetism. The quite contradictory results of these works were recently analyzed by Th. Dietl in his *Nature Mater.* review paper [2]. Th. Dietl summarized that not only the originally supposed interaction of "magnetic" atoms diluted in semiconducting oxide can be the source of experimentally observed RT ferromagnetism in ZnO and other oxides. In particular, it could be the non-uniformity of distribution of doping atoms, or the uncompensated spins in antiferromagnetic nanoparticles can generate weak ferromagnetic-like signal. The presence of unexpected high-temperature ferromagnetism in various oxides and carbon derivatives can be due to the magnetic moments residing on non-magnetic defects or impurities rather than on open d shells of transition metals [2–23]. In order to elucidate the dependence of room temperature (RT) ferromagnetism on the microstructure in ZnO, we have recently analyzed a large series of experimental publications with respect to

the present specific grain boundary area, i.e. the ratio of grain boundary (GB) area to grain volume s_{GB} [24]. FM only appears, if s_{GB} exceeds a certain threshold value s_{th} . This fact is broadly recognized now, in particular the RT ferromagnetic behaviour of pure and doped ZnO is explained by the oxygen vacancies or defects arranged in grain boundaries, interfaces or surfaces [7,9,14,16–19,21–23,25–28]. Based on this important finding, nano-grained pure and Mn-doped ZnO films have been prepared by us, which reveal reproducible RT-FM, where the magnetization is proportional to the film thickness, even for pure ZnO films [24,29]. Our findings strongly suggested that grain boundaries and related vacancies are the intrinsic origin for RT ferromagnetism. Ferromagnetic pure and doped ZnO is one of the most promising candidates for the application in spintronics. Therefore, this paper is devoted to the direct investigation of the structure of GBs in ZnO using high-resolution transmission electron microscopy (TEM).

2. Experimental details

ZnO thin films consisting of dense equiaxial nanograins were produced by using the novel method of liquid ceramics [30]. Zinc (II) butanoate diluted in the organic solvent with zinc concentrations between 1 and 4 kg/m³ was used as a precursor. The butanoate precursor was deposited on polycrystalline Al foils. Drying at 100 °C in air (about 30 min) was followed by thermal pyrolysis in an electrical furnace (in air) at 550 °C. Similar method was recently proposed for zinc oleates [31]. The presence of other magnetic impurities as Fe, Co, Mn and Ni was measured by atomic absorption spectroscopy in a Perkin–Elmer spectrometer and electron-probe microanalysis

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(EPMA) and remained below 0.001 at.%. The films were transparent and sometimes with a very slight greenish furnish with thicknesses between 50 and 900 nm. The thickness was determined by means of electron-probe microanalysis (EPMA) and edge-on transmission electron microscopy (TEM). EPMA investigations were carried out in a Tescan Vega TS5130 MM microscope equipped by the LINK energy-dispersive spectrometer produced by Oxford Instruments. TEM investigations were carried out on a Jeol JEM-4000FX microscope at an accelerating voltage of 400 kV.

3. Results and discussion

In Fig. 1 the bright high-resolution TEM micrograph of the ZnO film is shown. Direct lattice resolution permits to see the crystalline ZnO grains; they have wurzite structure according to the data of electron diffraction. One of these grains shown in the middle of the micrograph is completely surrounded by the amorphous area. Other ZnO/ZnO GBs are also covered by the amorphous phase. Fig. 2 contains the magnetization curve for ZnO thin film. The film clearly shows the pronounced ferromagnetism indicated by the saturation of magnetization J (above the applied field ~ 0.5 T). The saturation magnetization corresponds to about $0.8 \cdot 10^{-3} \mu\text{Bohr/f.u.}$ and is, therefore, comparable with data obtained in [24]. The inset shows the central part of the magnetization curve for ZnO with coercivity about 0.015 T. Therefore, similar to our previous work [24] and the papers of other authors [32–51] we observe the ferromagnetic behaviour in the pure, undoped ZnO without any magnetic impurity.

We have previously found an enriched Mn concentration in the amorphous grain boundary region in the Mn doped ZnO analogous system [52,53]. Earlier we also have observed that the Mn and Co solubility in ZnO strongly increased with decreasing grain size [29,53]. The quantitative estimation led to the conclusion that, close to the bulk solubility limit, the thickness of a Co- or Mn-enriched layer is several monolayers in GBs and at least two monolayers in the free surfaces [29,53]. This estimation was based on the hypothesis that the Co- or Mn-rich layers are uniformly thick and continuously cover all GBs. However, TEM micrographs revealed that the Mn-rich layers among ZnO grains are non-uniform. They look like the fine areas of the second amorphous phase which wets (covers) some of the ZnO/ZnO grain boundaries. The ferromagnetic behavior was observed in case of ZnO nanograins embedded in the amorphous matrix doped by Al [47].

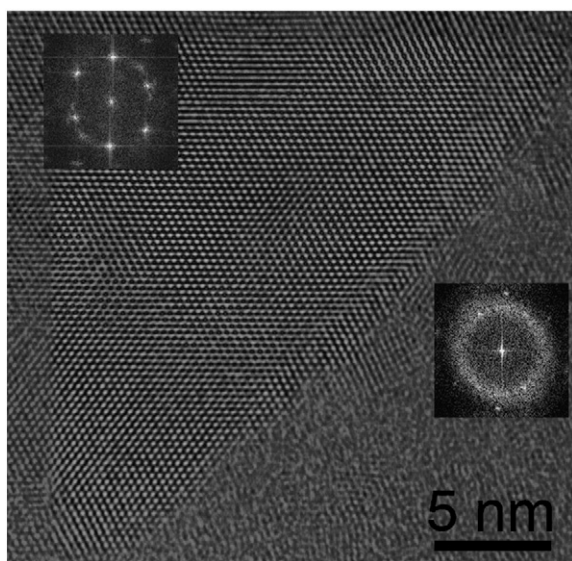


Fig. 1. Bright field high-resolution TEM micrograph of the ZnO film. Crystalline ZnO grains with wurzite structure are surrounded by the amorphous areas. Insets show the Fourier transformation pictures for the crystalline and amorphous areas.

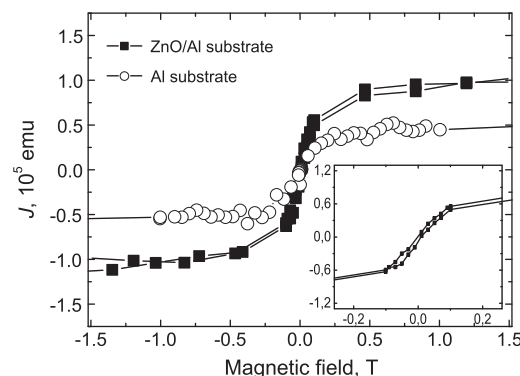


Fig. 2. Magnetization (calibrated in emu) at room temperature for ferromagnetic ZnO thin films (full squares). Open circles show the magnetization curve for the bare Al substrate. $0.8 \cdot 10^{-3} \mu\text{Bohr/f.u.}$ The inset shows the central part of the magnetization curve for ZnO.

However, in the latter case the ferromagnetic behavior appeared only above a certain Al concentration and not in pure ZnO.

The data obtained in [29,53] undoubtedly indicated that the Mn enrichment of GBs in fine-grained ZnO cannot be reduced to the simple single-layer GB segregation analysed by McLean [54]. Such layers of a GB phase of a finite thickness of few nm were first observed and theoretically treated with the aid of force-balance models in the pioneering works of David A. Clarke on silicon nitride [55,56]. Later, nanometer-thick, disordered films of a nearly constant or “equilibrium” thickness have been frequently observed in GBs in ceramics [57–62] and oxide/metal interfaces [63,64]. Thin equilibrium GB or surface films in the one-phase area of a bulk phase diagram were first considered by J.W. Cahn [65]. He proposed the idea that the transition from incomplete to complete surface wetting is a phase transformation. Later this idea was successfully applied for GBs, and also old data on GB wetting were reconsidered from this point of view [66–68]. GB wetting phase transformation proceeds at the temperature T_{wGB} where GB energy σ_{GB} becomes equal to the energy $2\sigma_{\text{SL}}$ of two solid/liquid interfaces. Above T_{wGB} GB is substituted by a layer of the melt. The tie-line of the GB wetting phase transition in the two-phase area of a bulk phase diagram continues into the one-phase area as a prewetting (or GB solidus) line. In the area between GB solidus and bulk solidus, GB contains the thin layer of a GB phase. The energy gain $(\sigma_{\text{GB}} - 2\sigma_{\text{SL}})$ above T_{wGB} permits to stabilize such thin layer of a GB phase, which is metastable in the bulk and become stable in the GB. The formation of metastable phase layer of thickness l leads to the energy loss $l\Delta g$. Finite thickness l of the GB phase is defined by the equality of the energy gain $(\sigma_{\text{GB}} - 2\sigma_{\text{SL}})$ and energy loss $l\Delta g$. In this simplest model, the prewetting GB layer of finite thickness l suddenly appears by crossing the prewetting (GB solidus) line $c_{\text{bt}}(T)$. Thickness l logarithmically diverges close to the bulk solidus. It is due to the fact that the thickness of a wetting phase is thermodynamically infinite in the two-phase area. Physically, in the two-phase area, its thickness is defined only by the amount of the wetting phase. Several ML thick liquid-like GB layers possessing high diffusivity were observed in the Cu–Bi [69], Al–Zn [70], Fe–Si–Zn [71] and W–Ni alloys [72].

However, the shape of the amorphous Mn-rich areas between ZnO grains [52] was very different for the very uniform and thin amorphous GB prewetting layers in the ZnO:Bi₂O₃ samples obtained by the liquid-phase sintering method [57–62]. They look like the fine areas of the second amorphous phase which completely surrounds some of the pure ZnO grains observed here and incompletely wets another ZnO/ZnO grain boundaries [52]. Similar morphology possess also the GBs in the undoped ZnO (Fig. 1). This structure is very similar to the situation when in a two-phase polycrystal the second phase completely wets some of the GBs and incompletely wets another GBs in the first phase [66–68]. Recently it was demonstrated that the

second (wetting) phase can be not only liquid, but also solid [73–75]. Fig. 1 reveals that the phase wetting the grain boundaries can also be amorphous, not only liquid or crystalline solid as in [66–68].

The wetting–prewetting phenomena discussed above are observed in the systems containing at least two components. It is easy to understand, why the GB wetting–prewetting layers appear in the ZnO doped by Mn [52] or Bi [57–62]. In order to understand the nature of the amorphous GB layers in pure ZnO we have to remember that – actually – it also contains two components, namely zinc and oxygen. The presence of the wetting-like amorphous zones between the wurtzite grains in the undoped ZnO could be the indication of the fact that these zones are enriched by one of the components (zinc or oxygen). The phenomenon of the high-temperature ferromagnetism in pure nanograined ZnO still remains miraculous [32–51]. It is frequently claimed to be driven by the defects [2–23,25–28,38–51]. The amorphous structure of intergranular layers and deficit of zinc or oxygen atoms can be the reason for the high-temperature ferromagnetism in pure nanograined ZnO.

As a result we can conclude that high resolution TEM reveals that the grain boundaries in the ferromagnetic nanograined ZnO films obtained by the “liquid ceramics” method are surrounded by amorphous layers. The morphology of these wetting amorphous layers is very different from that of the uniformly thin amorphous GB prewetting layers in the ZnO:Bi₂O₃ system and very similar to that in the Mn-doped ZnO. The presence of the wetting-like amorphous zones between the wurtzite grains in the undoped ZnO could be the indication of the fact that these zones are enriched by zinc or oxygen and, in turn, provoke the high-temperature ferromagnetism in pure nanograined ZnO.

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