ISSN 1063-7761, Journal of Experimental and Theoretical Physics, 2012, Vol. 115, No. 6, pp. 991–998. © Pleiades Publishing, Inc., 2012. Original Russian Text © K.P. Meletov, 2012, published in Zhurnal Eksperimental'noi i Teoreticheskoi Fiziki, 2012, Vol. 142, No. 6, pp. 1133–1141.

SOLIDS AND LIQUIDS

Phonon Spectrum and Interaction between Nanotubes in Single-Walled Carbon Nanotube Bundles at High Pressures and Temperatures

K. P. Meletov

Institute of Solid State Physics, Russian Academy of Sciences, Chernogolovka, Moscow oblast, 142432 Russia e-mail: mele@issp.ac.ru Received March 29, 2012

Abstract—The Raman spectra of single-walled carbon nanotubes at temperatures up to 730 K and pressures up to 7 GPa have been measured. The behavior of phonon modes and the interaction between nanotubes in bundles have been studied. It has been found that the temperature shift of the vibrational G mode is completely reversible, whereas the temperature shift of radial breathing modes is partially irreversible and the softening of the modes and narrowing of phonon bands are observed. The temperature shift and softening of radial breathing modes are also observed when samples are irradiated by laser radiation with a power density of 6.5 kW/mm². The dependence of the relative frequency Ω/Ω_0 for G^+ and G^- phonon modes on the relative change A_0/A in the triangular lattice constant of bundles of nanotubes calculated using the thermal expansion coefficient and compressibility coefficient of nanotube bundles shows that the temperature shift of the G mode is determined by the softening of the C–C bond in nanotubes. An increase in the equilibrium distances between nanotubes at the breaking of random covalent C–C bonds between nanotubes in bundles of nanotubes is in my opinion the main reason for the softening of the radial breathing modes.

DOI: 10.1134/S1063776112130079

1. INTRODUCTION

The effect of high temperatures and pressures on the structure and phonon spectrum of single-walled carbon nanotube bundles were studied using X-ray diffraction analysis and Raman spectroscopy [1-7]. The expansion of the triangular lattice of bungles of singlewalled carbon nanotubes at temperatures up to 950 K, which is due to the anharmonicity of the van der Waals interaction, as well as the radial expansion of nanotubes, was examined by the X-ray diffraction method [1]. The radial thermal expansion coefficient is very small, $\alpha_R = (-0.15 \pm 0.20) \times 10^{-5} \text{ K}^{-1}$, whereas the thermal expansion coefficient of the triangular lattice constant of bundles is relatively large, $\alpha_L = (0.75 \pm$ $(0.25) \times 10^{-5} \,\mathrm{K}^{-1}$. The radial thermal expansion of multiwalled carbon nanotubes and graphite in the direction along the graphene layers is very small [1]. At the same time, the temperature dependence of the distance between the walls of neighboring nanotubes is determined by the coefficient $\alpha_g = 4.2 \times 10^{-5} \text{ K}^{-1}$, which is larger than the thermal expansion coefficient of graphite in the direction perpendicular to the graphene layers, $\alpha_{gr} = 2.6 \times 10^{-5} \text{ K}^{-1}$ [1]. These features of the temperature behavior are due both to the strong covalent bond between carbon atoms in graphene layers and shells of nanotubes and to the weak van der Waals interaction between nanotubes in bundles and graphene layers in graphite [1].

Adsorbents, in particular, atmospheric gases play an important role in the temperature behavior of single-walled carbon nanotube bundles. The solution of molecular oxygen and nitrogen in channels between nanotubes in single-walled carbon nanotube bundles leads to a slight increase in the lattice constant of single-walled carbon nanotube bundles [8]. The temperature dependences of the lattice constant of singlewalled carbon nanotube bundles at heating in vacuum are different for heating and cooling cycles: after the completion of the first temperature cycle, the lattice constant decreases [2]. In the subsequent cycles, the temperature dependences coincide, but the repetition of the experiment after the aging of samples in the atmosphere leads to the recovery of the dependence characteristic of the first cycle. This is due to the desorption of atmospheric gases dissolved in singlewalled carbon nanotube bundles: molecular oxygen and nitrogen at heating in vacuum that are dissolved in the channels between nanotubes are removed, but single-walled carbon nanotube bundles are again saturated with gases at the subsequent aging of the samples in the atmosphere [2].

The temperature shift of phonon bands for radial breathing modes and the *G* mode caused by the vibrations of carbon atoms along a C–C bond was studied by the Raman scattering method at temperatures up to 800 K in single- [3, 5] and double-walled [4] carbon nanotubes. The measurements reported in [3, 5] and

molecular dynamics calculations for single-walled carbon nanotubes [3] indicate a negative temperature shift of phonon modes, which is due to the weakening of both the C–C bond in nanotubes and the interaction between nanotubes in the single-walled carbon nanotube bundles because of the anharmonicity of the van der Waals interaction. The same temperature dependence of phonon modes was obtained for double-walled carbon nanotubes [4]. The calculations reported in [3] also indicate that the thermal expansion of nanotubes in the radial direction is insignificant and does not contribute to the shift of phonon bands in agreement with the X-ray diffraction results [1].

X-ray diffraction investigations of single-walled carbon nanotube bundles at pressures up to 13 GPa show that the triangular lattice in single-walled carbon nanotube bundles exists at least up to a pressure of 10 GPa, the pressure dependence of the lattice constant is reversible, and the initial parameters are recovered with a decrease in the pressure [6]. The pressure dependence of the parameter A of the two-dimensional triangular lattice of single-walled carbon nanotube bundles is described by the one-dimensional analog of the equation

$$\frac{A}{A_0} = \left(\frac{\beta'}{\beta_0}P + 1\right)^{-1/\beta'},\tag{1}$$

where $\beta_0 = 43 \pm 4$ GPa is the bulk modulus and $\beta' = 33 \pm 3$ is its derivative with respect to the pressure [6]. These values are close to the respective values ab initio calculated for the bulk compression modulus of single-walled carbon nanotube bundles [7].

An important feature of carbon nanotubes is their capability of forming bundles where the coupling between individual nanotubes is so strong that their separation requires special solvents with surfactants and activation of the solution process by ultrasound [9]. The numerical calculations show that the van der Waals interaction between nanotubes is several times stronger than a similar interaction between fullerene molecules recalculated to the equivalent length of a nanotube [10]. The total interaction between them is strong because of their large length, which leads to the formation of bundles of nanotubes closely packed in a hexagonal lattice.

Another possible reason for the strong coupling between nanotubes in bundles can be random covalent C–C bonds between neighboring nanotubes [11]. These bonds appear owing to a large number of unsaturated double C=C bonds in nanotubes as this occurs in C₆₀ fullerene crystals under the action of light or at thermobaric treatment [12–14]. The ab initio numerical calculations predict the possibility of the formation of one- and two-dimensional structures, where neighboring nanotubes are coupled with each other by covalent bonds; some of these structures are more stable than the two-dimensional hexagonal lattice formed by nanotubes in single-walled carbon nanotube bundles [15]. The formation of such structures implies the deformation of nanotubes in the cross section and requires the application of high pressure [15].

The experiments show that, at certain parameters of thermobaric treatment, irreversible changes occur in the Raman spectra and the structure of singlewalled carbon nanotube bundles, which can be attributed to the formation of covalent bonds between nanotubes and carbon atoms with sp^3 coordination [16]. The formation of random covalent bonds between nanotubes in single-walled carbon nanotube bundles is also possible under the action of light as this occurs at the photopolymerization of C_{60} fullerene. It is known that the thermal treatment of polymers of C_{60} fullerene at a temperature of about 550 K leads to the destruction of polymer bonds and irreversible changes in the crystal structure and Raman spectra of the material [17, 18]. Similar effects can also be expected at the high-temperature annealing of single-walled carbon nanotube bundles if random covalent bonds between nanotubes are indeed formed.

In this work, the Raman spectra of carbon nanotubes were measured at temperatures up to 730 K, pressures up to 7 GPa, and intense laser irradiation in order to examine the phonon spectrum and the interaction between nanotubes, as well as possible transformations in single-walled carbon nanotube bundles. It is found that the temperature and pressure shifts of the G vibrational mode are completely reversible, whereas the temperature shift of the radial breathing modes is partially irreversible and cooling is accompanied by the softening of the modes and narrowing of phonon bands. A similar effect is observed with an increase in the laser radiation power density up to 6.5 kW/mm^2 . The dependence of the relative frequency Ω/Ω_0 for the G^+ and G^- phonon modes on the relative change in the triangular lattice constant A_0/A of single-walled carbon nanotube bundles is determined under the compression and expansion of the lattice and shows that the softening of the covalent bond in nanotubes makes the overwhelming contribution to the temperature shift of the G mode.

2. EXPERIMENTAL PROCEDURE

Carbon nanotubes were synthesized by the arc discharge method in the helium atmosphere at a pressure of 0.86 bar using the Ni/Y catalyst. The weight fraction of single-walled carbon nanotubes in the purified reaction product was approximately 90%; 2–4- μ m graphite microparticles constituted the main impurity, whereas the impurity of metals did not exceed 1.5%. The final product was annealed for 5 h in vacuum at 900 K and was single-walled carbon nanotube bundles in the form of flakes with sizes up to 100 μ m.

Individual flakes of single-walled carbon nanotubes with the characteristic size from 50 to 100 μ m were used for the measurements. The Raman spectra were measured with an Acton Spectra Pro-2500i spectrograph with a Pixis2K cooled detector. An Olympus



Fig. 1. Raman spectra of single-walled carbon nanotube bundles near the radial breathing modes and *G* mode: (a) initial single-walled carbon nanotubes after annealing at 530 K, and (c) single-walled carbon nanotubes after annealing at 630 K. The Raman spectra were measured at the same place of the sample under normal conditions with a laser radiation power density of 0.4 kW/mm^2 .

microscope and a 50× objective with a working distance of 14 mm were used to focus laser radiation on the sample. The cutoff of laser radiation was performed using a notch filter with a suppression depth of 10^{-3} in the band with a width of 120 cm⁻¹ near $\lambda =$ 532 nm. The laser beam was focused into a spot with a diameter of about 3 µm on the sample and the laser radiation power was approximately 5 mW.

The high-pressure measurements were performed with a high-pressure Mao–Bell-type diamond anvil cell. The 1 : 4 ethanol–methanol mixture was used as a pressure-transfer medium. The pressure was calibrated using the R_1 luminescence line of ruby [19]. The high-temperature measurements were performed with a homemade optical cell and a Termodat temperature controller. The temperature on the working table of the cell was maintained in the range of 50–500°C with an accuracy of $\pm 2^{\circ}$ C.

3. RESULTS AND DISCUSSION

The Raman spectra of single-walled carbon nanotube bundles in the regions of radial breathing modes and the *G* vibrational mode under normal conditions are shown in Fig. 1a. The *D* band whose intensity increases with the defectiveness of the graphite and carbon nanotube samples [20, 21] is not shown in the spectrum. The intensity of the *D* band in our samples was no more than 1% of the integral intensity of the *G* band, which indicates a fairly high structural perfection of single-walled carbon nanotubes [22]. The intense *G* band corresponds to the C–C vibrations of carbon atoms in the directions perpendicular and parallel to the axis of a nanotube (G^- and G^+ components corresponding to the bands near 1568 and 1591 cm^{-1} . respectively). The shape of the G^{-} band is determined by the electronic properties of the single-walled carbon nanotubes: a symmetric Lorentzian contour of the G^- band indicates semiconducting nanotubes, whereas an asymmetric Breit-Wigner-Fano contour indicates metallic nanotubes [23]. The most intense band in the low-frequency range with a frequency of 170 cm^{-1} corresponds to the radial breathing modes; satellites at frequencies of about 179 and 160 cm⁻¹ are located on both sides of this band. The frequency ω_R of phonons corresponding to the radial breathing modes in single-walled carbon nanotube bundles is inversely proportional to the diameter d_t of the nanotube and is described by the empirical formula [23]

$$d_t \text{[nm]} = 234 \text{ nm cm}^{-1} / (\omega_R \text{[cm}^{-1}] - 12 \text{ cm}^{-1}).$$
 (2)

According to Eq. (2), the main peak at 170 cm^{-1} corresponds to nanotubes with a diameter of about 1.48 nm, whereas 160- and 179-cm⁻¹ satellites correspond to nanotubes with diameters of about 1.58 and 1.40 nm, respectively. Figures 1b and 1c show the Raman spectra under normal conditions after the 30-min annealing of single-walled carbon nanotubes at temperatures of 530 and 630 K, respectively. After annealing, the radial breathing modes are shifted towards lower energies and are narrowed; the shift of the bands is larger for nanotubes with a larger diameter, which leads to a better resolution of the satellite bands. The shift of the bands increases with the annealing temperature and is maximal at 630 K. As can be seen in Figs. 1b and 1c, the annealing of the



Fig. 2. Raman spectra of single-walled carbon nanotube bundles near the radial breathing modes under normal conditions at the same place of the sample for the laser radiation power densities of (a) 0.4, (b) 1.7, (c) 6.5 kW/mm^2 , and (d) return to 0.4 kW/mm^2 .

samples does not lead to any changes in the *G* mode and the frequencies of the G^- and G^+ components remain unchanged within the measurement accuracy. It is noteworthy that these measurements were performed at a relatively low laser radiation power density of about 0.4 kW/mm². An increase in the power density to 1 kW/mm² insignificantly affects the Raman spectrum, but the subsequent increase leads to a decrease in the phonon frequencies, which is particularly pronounced at the maximum laser radiation power density of 6.5 kW/mm².

A decrease in the power density to the initial value of 0.4 kW/mm^2 leads to the recovery of the initial spectrum of phonons in the regions of the G^- and G^+ modes, but the frequencies of the radial breathing modes are not recovered and decrease noticeably. The Raman spectra for the single-walled carbon nanotubes for laser radiation powers from 0.4 to 6.5 kW/mm^2 are shown in Fig. 2, where the softening of the radial breathing modes after a decrease in the laser power to the initial value is seen. It is noteworthy that a change in the spectrum is observed only inside the laser spot, whereas changes in the neighboring dark regions are absent. It can be assumed that the dependence of the Raman spectra on the laser radiation power density is attributed to the heating of the sample inside the laser



Fig. 3. Temperature dependences of the frequencies of the radial breathing modes for single-walled carbon nanotube bundles under (open symbols) heating and (closed symbols) cooling. The circles, squares, and triangles correspond to the first (300–430 K), second (300–530 K), and third (300–730 K) annealing circles, respectively.

spot and the observed effect is similar to the heating of samples inside an optical cell.

The temperature dependences of the phonon frequencies of the radial breathing modes obtained by the Lorentzian approximation of the experimental contours of the bands are shown in Fig. 3. The measurements were performed at the same point of the sample in three successive heating-cooling cycles; the respective results are shown by various symbols. The temperature steps for heating and cooling were 25 and 50 K, respectively. The results indicate the irreversibility of the temperature dependence of phonon frequencies and the residual softening of the radial breathing modes after the cooling of the samples to the initial temperature. The softening of the modes occurs in the first temperature cycle (300-430 K, circles) and continues in the second temperature cycle (300–530 K, squares); the softening of the modes is almost absent only in the third temperature cycle (300-700 K, triangles) and the temperature dependence becomes reversible.

Figure 4 shows the temperature dependences of phonon frequencies for the G mode in the same three heating—cooling cycles. In contrast to the case of the radial breathing modes, the temperature dependences for the G mode are reversible within the measurement accuracy. It is also noteworthy that the intensity of the



Fig. 4. Temperature dependences of the frequency of the *G* mode for single-walled carbon nanotube bundles. The notation is the same as in Fig. 3.

D band remains almost unchanged after the annealing of samples at the maximum temperature of 730 K, because the defectiveness of nanotubes does not increase even after a fairly hard thermal treatment of single-walled carbon nanotube bundles.

A decrease in the frequency of the radial breathing modes after the annealing of single-walled carbon nanotube bundles depends on the temperature. This dependence was studied in a special series of measurements with individual samples in each annealing cycle. To this end, the Raman spectra were measured under normal conditions at a fixed point of the samples before and after their annealing. Annealing was performed during 20 min at temperatures up to 730 K. Softening was determined from the difference between the frequencies of phonons in two spectra. Figure 5 shows the resulting dependences for nanotubes with the diameters of 1.48 and 1.58 nm. The softening of the radial breathing modes is small up to an annealing temperature of 430 K, but increases rapidly with the temperature and is saturated at 530 K. With a further increase in the annealing temperature, the softening of the modes increases insignificantly and the dependence is of a quasi-threshold character with the threshold in the temperature interval of 430-530 K. It is also noteworthy that the samples after thermal treatment are quite stable in air because the subsequent measurements of the Raman spectra at room temperature for a week did not reveal noticeable changes.



Fig. 5. Softening of the radial breathing modes versus the temperature of annealing of single-walled carbon nanotube bundles with the diameters of $(\bigcirc$ 1.48 and (\bullet) 1.58 nm. The sharp softening of the radial breathing modes is observed in the shaded temperature interval.

According to the molecular dynamics calculations of the dynamics of single-walled carbon nanotube bundles, the effect of the van der Waals interaction on the phonon spectrum of nanotubes decreases with an increase in the frequency of vibrations and becomes negligibly small at frequencies above 500 cm⁻¹ [24]. This means that an increase in the distances between nanotubes should lead to a more noticeable decrease in the frequency of the radial breathing modes for nanotubes with a larger diameter, which have a relatively low frequency of the radial breathing modes; this behavior is observed in our experiments. At the same time, the reversible temperature shift of the G mode is comparable in absolute value with the shift of the bands of the radial breathing modes, although the contribution from the van der Waals interaction for these phonons is negligibly small [24]. As will be shown below, a decrease in the frequency of the Gmode is primarily due to the softening of C-C bonds under the heating of nanotubes.

In order to understand the reason for the irreversible decrease in the frequencies of the radial breathing modes under heating, we consider the possible mechanisms of this phenomenon. The softening of the radial breathing modes at the annealing of singlewalled carbon nanotubes can be attributed to an increase either in the diameters of the nanotubes or in the triangular lattice constant. According to X-ray dif-



Fig. 6. Frequency shift $\Omega - \Omega_0$ versus the (open symbols) temperature and (closed symbols) laser radiation power density for the (squares) G^- and (circles) G^+ modes.

fraction studies, the diameter of the nanotubes is almost independent of the temperature [1]. The irreversible increase in the diameters of the nanotubes was observed only under special conditions: the heating of single-walled carbon nanotube bundles to approximately 1800 K in the hydrogen atmosphere is accompanied by the joining of neighboring nanotubes with the doubling of their diameter [25]. This is obviously not the case in our experiments, where the annealing temperature, as well as a change in the frequency of a phonon (an "increase" in the diameter of the nanotube), is much smaller.

A change in the interaction between the nanotubes can be due to the removal of N₂ and O₂ dissolved atmospheric gases at the annealing of single-walled carbon nanotube bundles in vacuum [2]. The triangular lattice constant A of single-walled carbon nanotube bundles decreases irreversibly from 17.1 to 16.9 Å after the first heating-cooling cycle where dissolved gases are removed; the subsequent heating-cooling cycles lead to a reversible change in the lattice constant [2]. This occurs in the temperature interval of 300–600 K, i.e., approximately in the same temperature interval, where we measured the Raman spectra. However, it is noteworthy that a decrease in the lattice constant of single-walled carbon nanotube bundles should lead to the enhancement of the interaction between the nanotubes and to an increase in the frequencies of the radial breathing modes, whereas a decrease in the frequencies of the radial breathing modes can be attributed to the reverse process, i.e., to an increase in the lattice constant. It can be hypothesized that molecules dissolved in single-walled carbon nanotube bundles can enhance the van der Waals interaction between the nanotubes and their removal leads to the weakening of this interaction. However, it is senseless to discuss this, because there are no calculations of the interaction for this case.

The softening of the radial breathing modes can also be attributed to an increase in the distances between the nanotubes at the destruction of random covalent C–C bonds between them. Such bonds can appear in the process of growth of single-walled carbon nanotube bundles or at the irradiation of the samples by light as this occurs in C₆₀ fullerene molecules [12–14] and they are quite easily destroyed at the annealing of the samples [17, 18]. The quasi-threshold character of the temperature dependence of the softening of the radial breathing modes can be due to the breaking of such bonds in the temperature interval of 430–530 K if they indeed exist in single-walled carbon nanotube bungles.

Information on the interaction between the nanotubes in bundles can also be obtained from the shift of the G phonon mode, which was studied in the Raman spectra measured with various laser radiation intensities, high temperature, and high pressure. Figure 6 shows the dependences of the frequency of the G mode $(\Omega - \Omega_0)$ on the temperature and laser radiation power density. Here, Ω_0 is the frequency of the G mode at room temperature and a power density of 0.4 kW/mm^2 and Ω is the frequency of this mode at various temperatures and the same laser radiation power density or at room temperature and various laser radiation power densities. It can be seen that all data are well approximated by a linear dependence. This means that an increase in the power density leads to a noticeable heating of the sample inside the illuminated spot and the G^+ and G^- bands are shifted identically with the variation of the temperature and power density. The temperature shift coefficients dE/dT for the G^- and G^+ bands are -0.0230 ± 0.0007 and $-0.0229 \pm$ $0.0002 \text{ cm}^{-1}/\text{K}$, respectively, and coincide with each other within the measurement accuracy. The parallel shift of the bands means that the diameter of the nanotubes remains almost unchanged with the temperature, because the frequency of the G^- band increases with the diameter and approaches the frequency of the G^+ band (in the limit of the planar graphene sheet, this is one band corresponding to the doubly degenerate E_{2g} oscillation).

Figure 7 shows the pressure dependences of the frequencies of the G^+ and G^- bands for the direct and inverse pressure variations. These dependences are completely reversible, exhibit linear segments up to a pressure of 3 GPa, and become sublinear at higher pressures. The pressure shift coefficients dE/dP in the linear segments are 7.11 ± 0.29 cm⁻¹/GPa and $6.65 \pm$ 0.22 cm⁻¹/GPa for the G^- and G^+ bands, respectively. Thus, the bands "approach" each other with an increase in the pressure and the accuracy of the mathematical separation of the bands decreases. It is noteworthy that a decrease in the slopes of the pressure



Fig. 7. Pressure dependences of the frequencies of the G^+ and G^- bands of nanotubes in bundles at room temperature at an (open symbols) increase and (closed symbols) decrease in the pressure.

dependences of the frequencies of the G^+ and $G^$ bands near a pressure of 2 GPa was observed in early works [26, 27], but more accurate measurements in a wide pressure range up to 40 GPa provide smooth sublinear dependences throughout the entire measurement interval [28].

The experimental compression modulus and thermal expansion coefficient of the triangular lattice of single-walled carbon nanotube bundles [1, 6] make it possible to determine the dependences of the relative frequency Ω/Ω_0 for the G^- and G^+ bands on the relative change of the triangular lattice constant A_0/A . The relative change of the triangular lattice constant A_0/A under compression was calculated by Eq. (1) with the data from [6] for the compression modulus $\beta_0 = 43 \pm$ 4 GPa and its derivative with respect to the pressure $\beta' = 33 \pm 3$. The expansion-induced change in the relative triangular lattice constant A_0/A was calculated using the thermal expansion coefficient $\alpha = (0.75 \pm$ $(0.25) \times 10^5 \text{ K}^{-1}$ [1]. The dependences for the expansion of the lattice (heating) and for the compression of the lattice (high pressure) are shown in the left lower and right upper parts of Fig. 8, respectively. The dependence of Ω/Ω_0 on A_0/A for the compression of the lattice is superlinear, as should be expected at the dominant contribution from the van der Waals inter-



Fig. 8. Relative frequency Ω/Ω_0 versus the relative triangular lattice constant A_0/A for the (squares) G^- and (circles) G^+ modes in nanotube bundles. The left lower segment corresponds to the expansion of the lattice at high temperatures, whereas the right upper segment corresponds to the compression of the lattice at high temperatures.

action between the nanotubes. The dependence of Ω/Ω_0 on A_0/A for the expansion of the lattice is linear in a relatively narrow range of the experimental data. It is noteworthy that the dependence in the equivalent range of the compression data can also be treated as linear, but its slope is several times larger than that for the compression of the lattice. This discrepancy indicates a significant contribution of the softening of covalent C–C bonds in nanotubes to the temperature shift of the *G* mode, which is much larger than the contribution from the anharmonicity of the van der Waals interaction.

To summarize, the results indicate that the overwhelming contribution to the temperature shift of the G mode comes from the softening of covalent bonds in nanotubes. The van der Waals interaction plays the decisive role in the pressure shift of the G mode and in the temperature shift of the radial breathing modes. In my opinion, the softening of the radial breathing modes after the annealing of the single-walled carbon nanotube bundles is attributed to an increase in the distances between nanotubes after the destruction of random covalent bonds between them.

ACKNOWLEDGMENTS

This work was supported by the Russian Foundation for Basic Research (project no. 11-02-00886) and by the Presidium of the Russian Academy of Sciences (program "Physics of Strongly Compressed Matter").

REFERENCES

- Y. Maniwa, R. Fujiwara, H. Kira, H. Tou, H. Kataura, S. Suzuki, Y. Achiba, E. Nishibori, M. Takata, M. Sakata, A. Fujiwara, and H. Suematsu, Phys. Rev. B: Condens. Matter 64, 241402 (2001).
- 2. Y. Yosida, Rigaku J. 19, 42 (2002).
- N. R. Raravikar, P. Keblinski, A. M. Rao, M. S. Dresselhaus, L. S. Schadler, and P. M. Ajayan, Phys. Rev. B: Condens. Matter 66, 235424 (2002).
- Z. Zhou, L. Ci, L. Song, X. Yan, D. Liu, H. Yuan, Y. Gao, J. Wang, L. Liu, W. Zhou, S. Xie, Y. Du, and Y. Mo, Chem. Phys. Lett. **396**, 372 (2004).
- H. D. Li, K. T. Yue, Z. L. Lian, Y. Zhan, L. X. Zhou, S. L. Zhang, Z. J. Shi, Z. N. Gu, B. B. Liu, R. S. Yang, H. B. Yang, G. T. Zou, Y. Zhang, and S. Iijima, Appl. Phys. Lett. **76**, 2053 (2000).
- S. M. Sharma, S. Karmakar, S. K. Sikka, P. V. Teredesai, A. K. Sood, A. Govindaraj, and C. N. R. Rao, Phys. Rev. B: Condens. Matter 63, 205417 (2001).
- 7. S. Reich, C. Thomsen, and P. Ordejon, Phys. Rev. B: Condens. Matter **65**, 153407 (2002).
- A. Fujiwara, K. Ishii, H. Suematsu, H. Kataura, Y. Maniwa, S. Suzuki, and Y. Achiba, Chem. Phys. Lett. 336, 205 (2001).
- M. J. O'Connell, S. M. Bachilo, C. B. Huffman, V. C. Moore, M. S. Strano, E. H. Haroz, K. L. Rialon, P. J. Boul, W. H. Noon, C. Kittrell, J. Ma, R. H. Hauge, R. B. Weisman, and R. E. Smalley, Science (Washington) 297, 593 (2002).
- 10. L. A. Girifalco, M. Hodak, and R. S. Lee, Phys. Rev. B: Condens. Matter **62**, 13104 (2000).
- A. Kis, G. Csányi, J. P. Salvetat, T.-N. Lee, E. Couteau, A. J. Kulik, W. Benoit, J. Brugger, and L. Forró, Nat. Mater. 3, 153 (2004).
- A. M. Rao, P. Zhou, K.-A. Wang, G. T. Hager, J. M. Holden, Y. Wang, W. T. Lee, X. X. Bi, and P. C. Elkund, Science (Washington) 259, 955 (1993).

- Y. Iwasa, T. Arima, R. M. Fleming, T. Siegrist, O. Zhou, R. C. Haddon, L. J. Rothberg, K. B. Lyons, H. L. Carter, Jr., A. F. Hebard, R. Tycko, G. Dabbagh, J. J. Krajewski, G. A. Thomas, and T. Yagi, Science (Washington) 264, 1570 (1994).
- M. Nunez-Regueiro, L. Marques, J.-L. Hodeau, O. Bethoux, and M. Perroux, Phys. Rev. Lett. 74, 278 (1995).
- 15. T. Yldirim, O. Gülseren, Ç. Kiliç, and S. Ciraci, Phys. Rev. B: Condens. Matter **62**, 12648 (2000).
- V. N. Khabashesku, Z. Gu, B. Brinson, J. L. Zimmerman, J. L. Margrave, V. A. Davydov, L. S. Kashevarova, and A. V. Rakhmanina, J. Phys. Chem. B 106, 11155 (2002).
- Y. Iwasa, K. Tanoue, T. Mitani, and T. Yagi, Phys. Rev. B: Condens. Matter 58, 16374 (1998).
- M. V. Korobov, V. M. Senyavin, A. G. Bogachev, E. B. Stukalin, V. A. Davydov, L. S. Kashevarova, A. V. Rakhmanina, V. Agafonov, and A. Szwarc, Chem. Phys. Lett. 381, 410 (2003).
- 19. A. Jayaraman, Rev. Sci. Instrum. 57, 1013 (1986).
- 20. F. Tuinstra and J. Koenig, J. Chem. Phys. 53, 1126 (1970).
- 21. R. J. Nemanich and S. A. Solin, Phys. Rev. B: Condens. Matter 20, 392 (1979).
- 22. C. Thomsen and S. Reich, Phys. Rev. Lett. 85, 5214 (2000).
- 23. U. D. Venkateswaran, Phys. Status Solidi B 241, 3345 (2004).
- 24. M. S. Dresselhaus and P. C. Eklund, Adv. Phys. 49, 705 (2000).
- 25. S. L. Fang, A. M. Rao, P. C. Eklund, P. Nikolaev, A. G. Rinzler, and R. E. Smalley, J. Mater. Res. 13, 2405 (1998).
- U. D. Venkateswaran, E. A. Brandsen, U. Schlecht, A. M. Rao, E. Richter, I. Loa, K. Syassen, and P. C. Eklund, Phys. Status Solidi B 223, 225 (2001).
- 27. S. Karmakar, S. M. Sharma, P. V. Teredesai, D. V. S. Muthu, A. Govindaraj, S. K. Sikka, and A. K. Sood, New J. Phys. 5, 143 (2003).
- A. Merlen, N. Bendiab, P. Toulemonde, A. Aouizerat, A. San Miguel, J. L. Sauvajol, G. Montagnac, H. Cardon, and P. Petit, Phys. Rev. B: Condens. Matter 72, 035409 (2005).

Translated by R. Tyapaev