

Raman spectra and calorimetric studies of MWCNTs

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Results

Calorimetric, DSC, XPS, NEAFS and Raman studies of multi-walled carbon nanotubes (MWCNTs) have been done. The experimental results are shown in Fig. 1-4. The heat capacity of MWCNTs in the temperature interval from 2 to 275 K was measured by the relaxation method using PPMS [1]. The size effects are observed in the temperature dependences of the specific heat of $C_p(T)$ for MWCNTs. The kinetic processes in MWCNTs were studied from room temperature to 500 K in a nitrogen flow using differential scanning calorimetry (DSC) on a Perkin - Elmer - 8000. It was found that all studied MWCNTs have a small number of defects, functional groups, and other impurities. The Raman measurements were performed in backscattering geometry using a Renishaw InVia Raman microscope equipped with a confocal DM 2500 Leica optical microscope and a CCD detector. In the Raman spectra of MWCNTs, defect-induced phonon mode so-called D band is observed near 1346 cm^{-1} . The high-energy G mode (1572 cm^{-1}), due to vibrations of carbon atoms in the walls of the nanotubes, is shifted toward the lower frequencies compared to the G mode of graphite (1580 cm^{-1}). The 2D band related with the two-phonon scattering is observed near 2688 cm^{-1} . The dependence of the I_D/I_{2D} ratio on the average diameter of MWCNTs is in good agreement with the data of [2]. $I_D > I_{2D}$ for MWNTs with an average diameter $d < 15\text{ nm}$ and $I_D < I_{2D}$ for MWNTs with $d > 15\text{ nm}$.

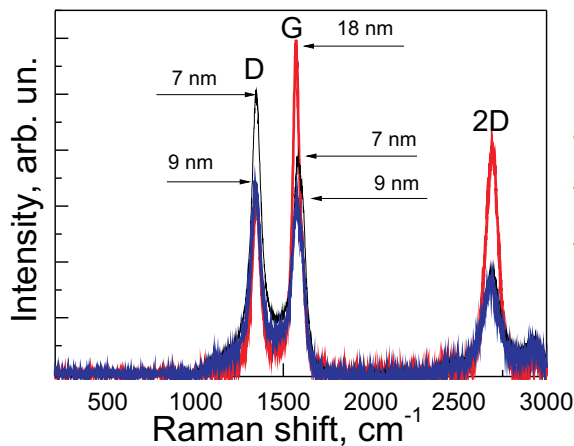


Fig. 1. Raman spectra for MWCNTs.

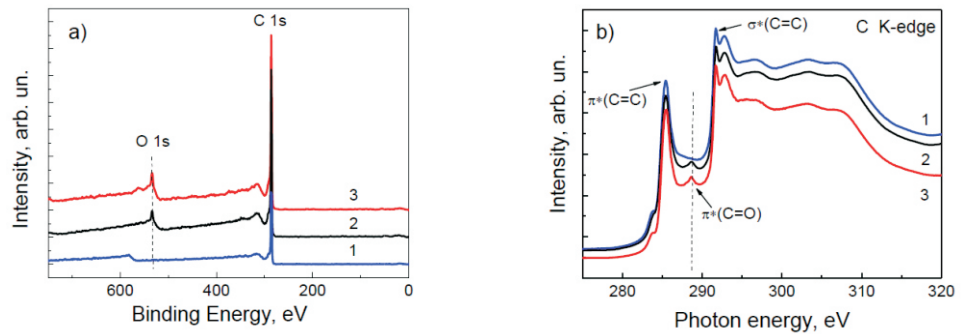


Fig. 4. XPS (a) and NEXAFS (b) spectra of initial (1), milled (2) and oxidized - then milled (3) multi-walled carbon nanotubes (18 nm).

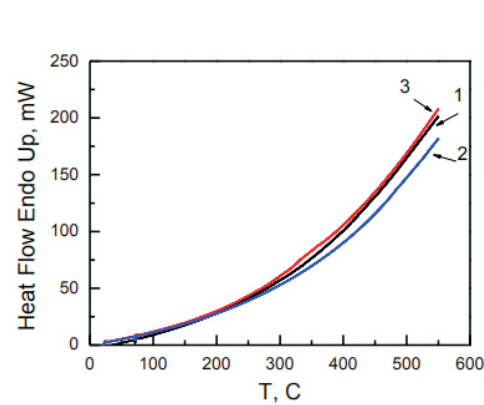


Fig. 2. DSC curves of MWCNTs with the average diameter 7 nm (curve 1), 9 nm (curve 2), and 18 nm (curve 3).

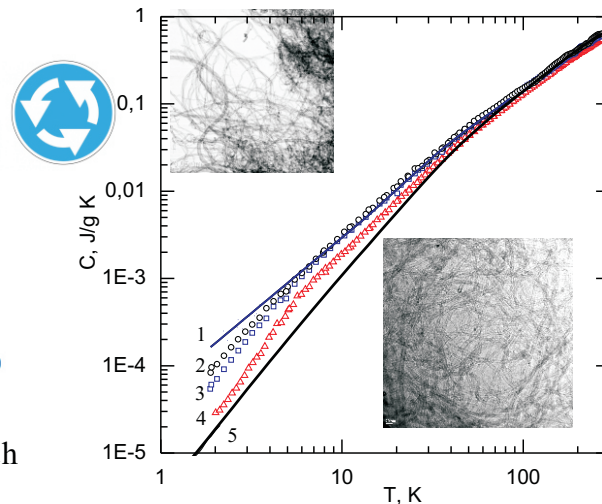


Fig. 3. The temperature dependences of the specific heat of the MWCNTs 7 nm (curve 2), 9 nm (curve 3) and 18 nm (curve 4) in comparison with specific heat of single-walled SWNT bundles (1) and graphite (5). The insert: TEM of MWCNTs 7 nm (up) and 18 nm (below).

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[1] V.V. Sumarokov, A Jeżowski, D Szewczyk, M.I. Bagatskii, M.S. Barabashko, A.N. Ponomarev, V.L. Kuznetsov, S.I. Moseenkov, *Low Temp. Phys.* **45**(3), 347–354, (2019). [*Fiz. Nizk. Temp.* 45(3), 395–403 (2019)].

[2] S.N. Bokova-Sirosh, V.L. Kuznetsov, A.I. Romanenko, M.A. Kazakova, et al., *J. Nanophotonics* 10, 012526 (2016).