FULLERENES, NANOTUBES, AND CARBON NANOSTRUCTURES
Vol. 12, Nos. 1 & 2, pp. 293–296, 2004

Infrared Spectra of Hydrogenated C_{60}, Carbon Nanotubes, and Nanofibers

A. V. Bazhenov,* T. N. Fursova, V. E. Antonov, I. O. Bashkin, A. L. Harkunov, and E. G. Ponyatovsky

Institute of Solid State Physics, Chernogolovka, Moscow, Russia

ABSTRACT

Hydrogenated C_{60}, carbon single-wall nanotubes (SWNT) and nanofibers (NF) were studied by means of IR transmittance spectroscopy of polycrystalline samples, of powder pressed in KBr pellets (TPKBr) and diffuse reflection (DR). Diffuse reflection was found to be a preferable method. Hydrogenation reduces the high-frequency conductivity σ of the free carriers by 10 and 30 times in SWNT and NF, respectively, and results in the appearance of a C–H vibrational mode. This mode disappears and σ restores partially after an annealing of the samples at 700°C.

Key Words: Nanotubes; Nanofibers; Infrared spectra; Hydrogenated C_{60}; Single-wall nanotubes.

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*Correspondence: A. V. Bazhenov, Institute of Solid State Physics, Chernogolovka, Moscow 142432, Russia; E-mail: bazhenov@issp.ac.ru.

DOI: 10.1081/FST-120027182
1536-383X (Print); 1536-4046 (Online)

Hydrogenation of single-wall nanotubes (SWNT) and nanofibers (NF) at a rather small pressure ≤ 0.1 Kbar and temperatures $T = 77 \div 300$ K showed that the dominant mechanism of hydrogen absorption was the physisorption of H_2 molecules, the desorption taking place at $T \leq 300$ K.[1] We produced hydrogenated NF and SWNT at high pressure (30–90 Kbar) by the method described elsewhere.[2] They contained 6–7 wt% of hydrogen and differed from known hydrogenated nanostructures by thermal stability: most of hydrogen was released at $t \geq 500°C$.

IR spectra were measured at $T = 300$ K, from 400 to 5000 cm^{-1}, by a Fourier-spectrometer. Mechanical treatment of the hydrogenated thick

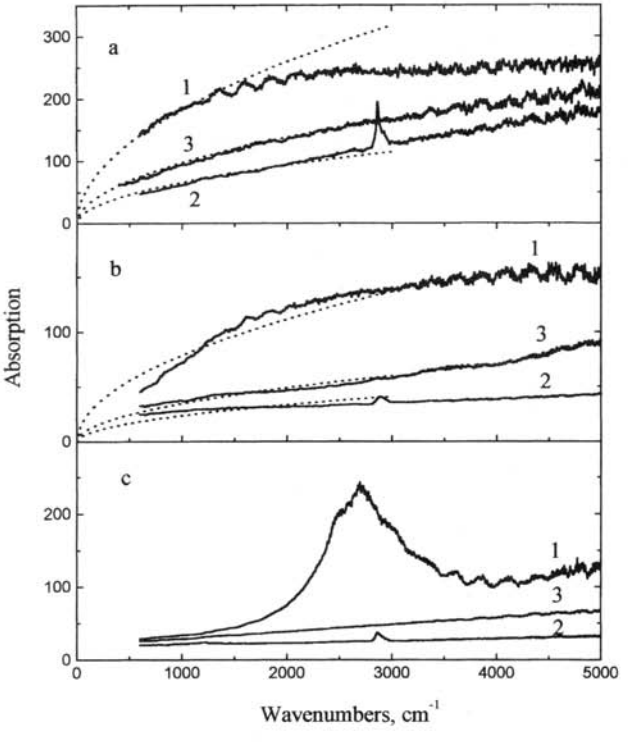

Figure 1. Absorption spectra K restored from diffusive reflection R by using the Kubelka—Munk equation $K = (1 - R)^2 S/2Rc$, where S is the scattering coefficient, concentration $c = 1$ in our case. Spectra 1, 2, and 3 are initial nanostructures, hydrogenated and annealed at 700°C during 6 hr, respectively. (a) Spectra of NFs, (b) and (c) SWNT with admixture of the graphite nanoparticles, $p \cong 10\%$ and 50%, respectively. Dashed curves are the Drude approximation of the absorption spectra, $K = A^* sqrt(v)$ at wavenumbers $v \ll \gamma$, γ—damping factor of free carriers.

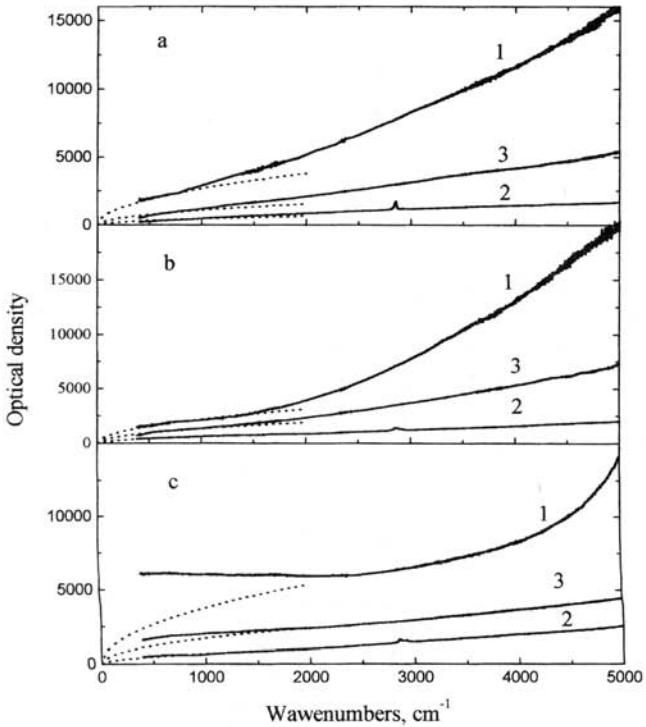

Figure 2. Absorption spectra K of powders pressed in KBr pellets: (a) NFs, (b) and (c) SWNTs with 10% and 50% admixture of the graphite nanoparticles, respectively. K was calculated from the tramsmittance spectra T as $K = -\ln(T)$. All other notations are analogous to those ones of Fig. 1.

samples can result in a hydrogen release. So, we cleaved samples into thin plates. Absorption lines associated with vibrational modes of the C–H bonds were well detected in the transmittance spectrum of $C_{60}H_{36}$ polycrystalline plates with the help of IR microscope. This method was not applicable, however, for the study of SWNTs and NFs due to the free carrier absorption and strong variation of the plate thickness. The grinding of $C_{60}H_{36}$ does not result in a loss of hydrogen. We have assumed that grinding is applicable for both NFs and SWNTs as well.

The comparison of the absorption spectra calculated from DR of powder (Fig. 1) and TPKBr (Fig. 2) shows their qualitative concurrence. The spectra of hydrogenated NFs and SWNTs distinguish from the spectra of plates by a line 2900 cm^{-1} (stretching C–H vibration). Monotonic growth of absorption

with wavenumber is determined by the conductivity of the free carriers σ. For both methods the Drude approximation of the low-energy range of the spectra showed that hydrogenation decreases σ of SWNTs, $\sigma_0/\sigma_H = 10$. In the case of NFs, σ_0/σ_H was evaluated as 8 and 32 from the spectra of Figs. 1 and 2, respectively. However, the DR method is preferable for the investigation of nano-objects because it neither destroys samples nor demands their special preparation. The annealing of the samples results in disappearance of the C–H modes. The free-carrier absorption restores partially, probably due to incomplete removal of hydrogen.

It is worth noting that DR method is sensitive to the presence of graphite nanoparticles. Their $p \cong 50\%$ admixture in the SWNTs resulted in appearance of the "absorption" line at 2700 cm^{-1} in the DR spectrum 1 of Fig. 1(c) and in additional absorption below 2500 cm^{-1} in the spectrum 1 of Fig. 2(c). These features were absent in the spectra of SWNTs with $p \cong 10\%$ [Figs. 1(b) and 2(b)].

ACKNOWLEDGMENTS

The work was supported by RFBR grant No. 02-02-16859, RAS Program "New materials and structures" and Russian R&D Program "Fullerenes and atomic clusters."

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Submitted July 4, 2003
Accepted August 10, 2003