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Magnetoresistance of functionalized carbon nanotubes

Der magnetoresistive Effekt von funktionalisierten Kohlenstoffnanoröhren

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In present work a novel method for the functionalization of carbon nanotubes is proposed. The magnetoresistance of carbon nanotubes specimens in temperature range from 1.6 K up to 85 K and in magnetic field up to 5 T was investigated. It was shown that the proposed functionalization method does not cause any new defects in structure of carbon nanotubes and does not essentially influence the resistivity of nanotubes. It is revealed the appearance of the charge carriers weak localization and interactions effects for as-prepared and functionalized carbon nanotubes. On the basis of the experimental data, the explicit type of temperature dependence of wave function phase relaxation time and Fermi-level energy value for as-prepared and functionalized carbon nanotube are established.

Keywords: Carbon nanotubes / functionalization / weak localization / charge carriers interaction

Schlüsselwörter: Kohlenstoff-Nanoröhren / Funktionalisierung / schwache Lokalisierung / Ladungsträger-Interaktion

1 Introduction

It is well known that carbon nanotubes (CNTs) do not form dispersions or solutions in organic solvents and water. Carbon nanotubes tend to aggregate and form bundles and matted nets due to the relatively strong Van der Waals long-range interaction that occurs between separated carbon nanotubes. However, the implementation of a stable homogeneous dispersion of carbon nanotubes in a polymer matrix is the most important issue in creation of polymer composites with homogeneous structure. When carbon nanotubes interact with some substances such as oxygen, fluorine, ozone, oxygen-containing acids and salts, water-soluble polymers some functional groups join nanocarbon surface. These functional groups modify the carbon nanotubes surface and create the conditions for homogeneous distribution of carbon nanotubes filler in a polymer matrix and for strong bond between tubes and polymer matrix [1–3]. The largest number of functional groups on surface of the carbon nanotubes occurs through oxidation [4, 5], fluorination and amidation (so-called covalent functionalization [6, 7]. It is generally accepted that covalent functionalization results in increased degree of defects in the tubes structure and destruction of delocalized π electron system of gra-

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phite layers. This obviously leads to degradation of tubes properties associated with charge transfer. Thus, on one hand, the functionalization of carbon nanotubes is an essential process that provides uniform distribution of carbon nanotubes in the polymer matrix and, on the other hand, it leads to disruption of the transport properties of the carbon nanotube. Therefore, the question that arises is whether such possible functionalization methods allow the modification of the surface of carbon nanotubes, while not restricting their transport properties. Many published works dealing with various methods for covalent functionalization of carbon nanotubes appear in literature [8–13]. However, in these studies the impact of the carbon nanotubes functionalization methods on their transport properties was not investigated. Consequently, the aim of the present work is the investigation of influence of carbon nanotubes functionalization method on resistivity of nanotubes. Furthermore, the changes of carbon nanotube's electron structure parameters at functionalization were investigated based on low-temperature studies of magnetoresistance.

2 Experimental

For our investigations two types of carbon nanotubes were used. As-prepared carbon nanotubes were produced by means of chemical vapour deposition (CVD) method with using nickel catalyst. More details concerning the structure characterization of as-prepared and functionalized carbon nanotubes by scanning electron microscopy (SEM), X-ray diffraction (XRD), as well as results of investigations of functional groups qualitative composition on the surface of carbon nanotubes are presented in previously published paper [14]. According to electron microscopy data the outer diameter of carbon nanotubes varies from 10 nm to 20 nm while their length is about 15 μm, *Fig. 1*. The functionalization of initial carbon nanotubes was carried out with a treatment with a mixture of concentrated H_2SO_4 and HNO_3 (volume ratio 3 : 1). The suspension of carbon nanotubes was dispersed on the magnetic stirrer for 1 h and then was boiled at T = 353 K for 4 h. The X-ray diffraction investigations of carbon nanotube specimens were performed using DRON-4-07 X-ray diffractometer (NiKα filtered radiation), the wavelength was λ = 0.165791 nm and the exposition time was

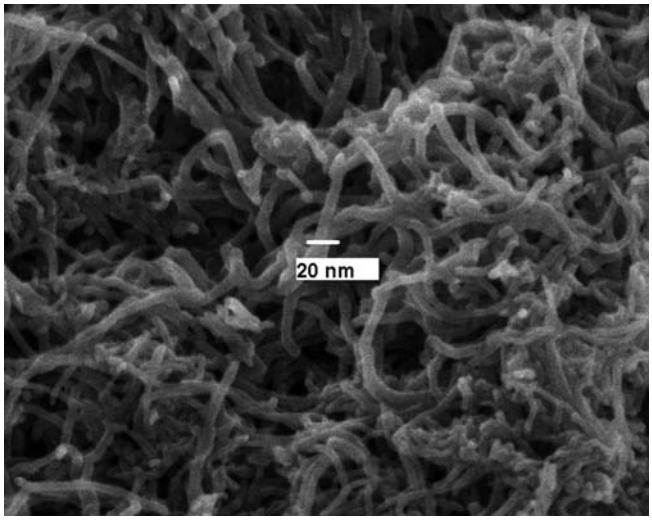

Figure 1. Fragment of scanning electron microscopy – image of as-prepared carbon nanotubes

equal to 6 s. The size of the coherent scattering region (the size of crystallite) was estimated from half-width B of 002 graphite line by using the following equation: $L = k\lambda/\beta \cos(\theta)$, $\beta = (B^2 - b^2)^{0.5}$ where k is the form-factor of the reflection, k = 0.89, b = 0.00239 radian the half-width of the standard reflection (we use pure natural graphite as standard). *Fig. 2* presents the fragments of X-ray diffraction pattern for the as-prepared and the functionalized carbon nanotubes. There is intensive 002-graphite line for as-prepared and functionalized carbon nanotubes that corresponds to interplanar distance d_{002} = 0.336 nm, Fig. 2. X-ray diffraction also contains low intensive nickel lines that reflecting the presence of catalyst's particles in carbon nanotubes. The intensity of these lines decreases after functionalized pro-

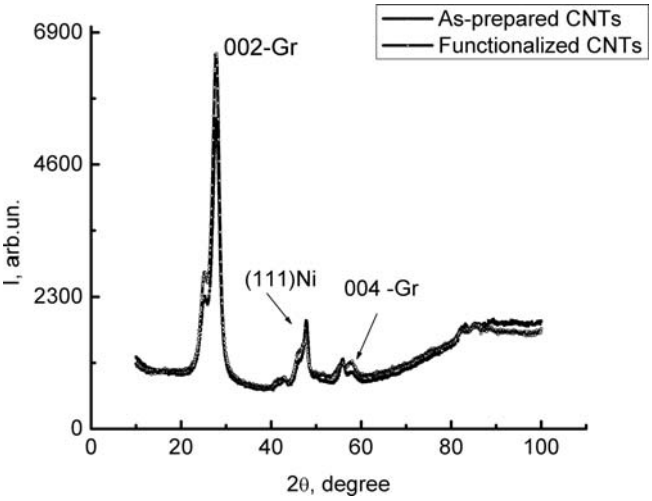

Figure 2. Fragments of X-ray diffraction pattern for as-prepared and functionalized carbon nanotubes

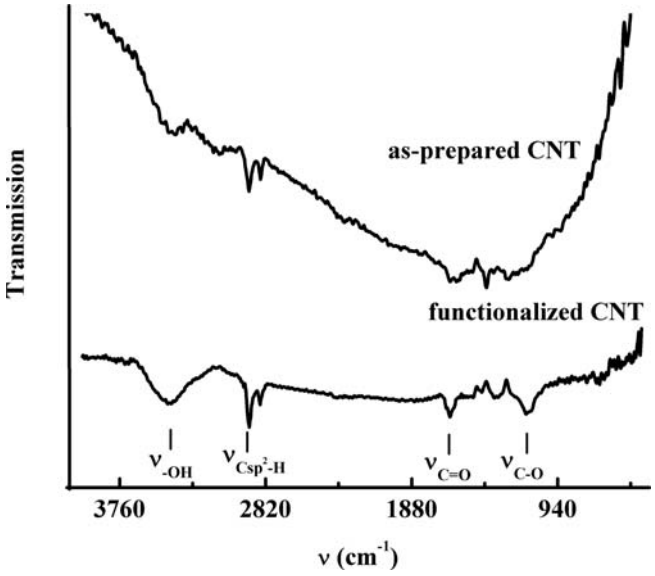

Figure 3. IR-spectra of as-prepared and functionalized carbon nanotubes

cess. The estimation of crystallite size gives the value of L ≈ 10 nm for as-prepared and functionalized carbon nanotube.

Investigations of the qualitative composition of the functional groups on the surface of functionalized carbon nanotube were performed using IR-spectroscopy method. The experiments were carried out by using a Perkin Elmer Spectrum BX FT-IR infrared spectrometer in the frequency range of 4000–400 cm^{-1} in transmission mode. The specimens in a form of pellets with 10 mm in diameter were prepared from the powder mixture of carbon nanotube and KBr. The pellets were carefully dried before measurements. The IR-spectra of as-prepared and functionalized carbon nanotubes are presented in *Fig. 3*. In IR-spectrum of as-prepared carbon nanotubes bands corresponding to stretching vibrations of –C–H-group (2852 cm^{-1}, 2924 cm^{-1}) are observed, Fig. 3. After functionalization of carbon nanotubes the new intensive bands corresponding to stretching vibrations of –OH (3430 cm^{-1}), >C=O (1636 cm^{-1}) and –C–O (1136 cm^{-1}) groups appear.

For measurements of magnetoresistance the compacted specimens of carbon nanotubes were prepared with method of cold pressing using a polyvinyl acetate adhesive (85 % mass of carbon nanotube and 15 % mass of polyvinyl acetate). The density of obtained compacted specimens was about 1.35 g/cm^3. Magnetoresistance of specimens was investigated in the temperature range from 1.6 K up to 85 K using the standard four-probe technique. The experimental errors for the measurements of electrical resistivity and resistance were estimated as 0.5 % and 0.05 %, respectively. The magnetic field induction up to 5 T was applied perpendicular to the direction of the electric current.

3 Results and discussion

Fig. 4 shows the dependence of resistivity on magnetic field for as-prepared carbon nanotube and for functionalized carbon nanotube specimens. The resistivity decreases with increasing magnetic field for as-prepared and functionalized carbon nanotubes in whole studied temperature range, Fig. 4. The experimental temperature dependence of magnetoresistance, defined as $\frac{\Delta\rho}{\rho} = \frac{\rho_B - \rho_0}{\rho_0}$, where ρ_B is the mag-

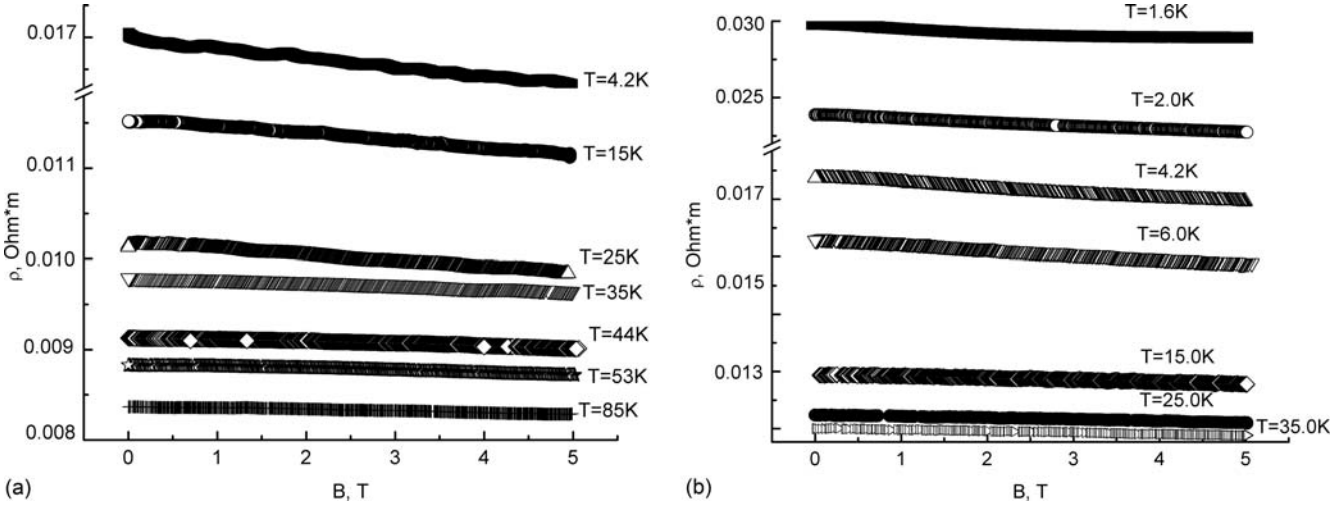

Figure 4. ρ(B) dependences for as-prepared carbon nanotubes (a) and for functionalized carbon nanotubes (b)

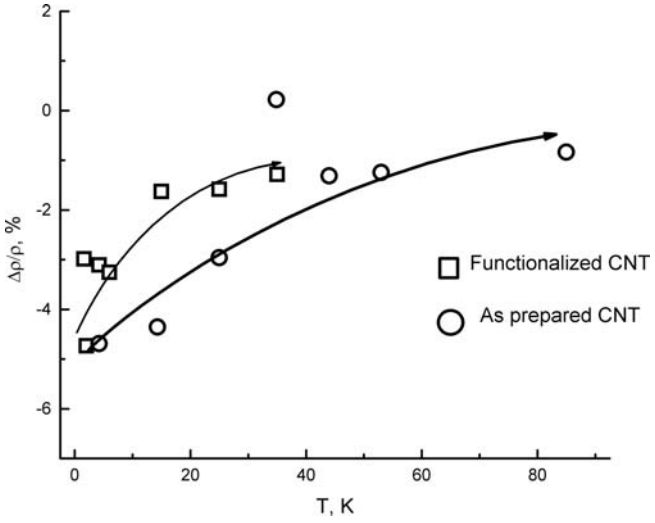

Figure 5. Δρ/ρ(T) dependences in the maximum magnetic field (B_{max} = 5 T) for as-prepared and functionalized carbon nanotubes

netoresistance in maximum magnetic field, ρ_0 the magnetoresistance in zero magnetic field, for as-prepared and functionalized carbon nanotubes are shown in *Fig. 5*. The maximum absolute values of magnetoresistance are 4.7 % for as-prepared carbon nanotubes and 3.5 % for functionalized carbon nanotubes, Fig. 5. With increasing temperature the absolute values of the negative magnetoresistance decrease but the magnetoresistance absolute value of as-prepared carbon nanotubes seems to be always greater compared to that of functionalized carbon nanotubes. *Fig. 6* shows the temperature dependences of resistivity in zero magnetic field for both carbon

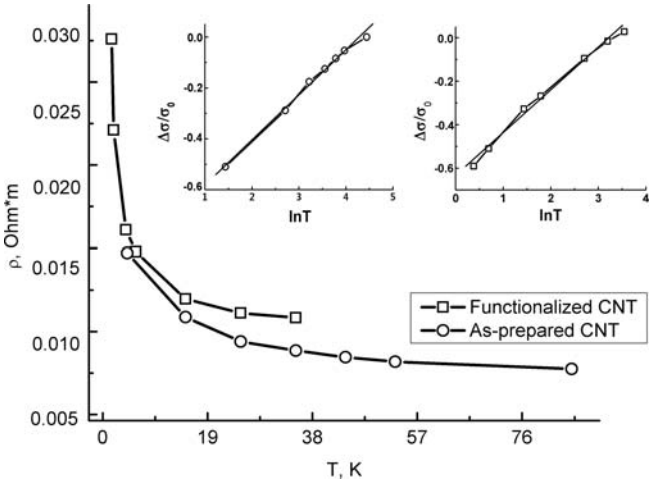

Figure 6. ρ(T) dependences in zero magnetic fields for as-prepared and functionalized carbon nanotubes, insets: dependence $\frac{\sigma(T)}{\sigma_0} - 1 = \frac{\Delta\sigma}{\sigma_0}$ vs. ln (T) for as-prepared and functionalized carbon nanotube

nanotubes specimens. The decrease of resistivity with the rise in temperature takes place for both as-prepared and functionalized carbon nanotubes specimens, but, in whole low temperature range the resistivity of functionalized carbon nanotubes is slightly higher compared to that of as-prepared carbon nanotubes, Fig. 6.

The properties of the carbon nanotubes, associated with charge transfer, were investigated, their kinetic properties can be described in the model of two-dimensional conduction [15–17]. For such weakly ordered systems, which are multiwall carbon nanotube at low temperatures, there are quantum effects of weak localization and interaction of charge carriers [15–18]. One of the characteristic features of these effects is negative magnetoresistance, the absolute value of which increases with decreasing temperature. This effect was also observed for carbon nanotubes specimens studied in present work. Another characteristic feature of weak localization and interaction effects is the irregular decrease of conductivity at low temperature. The associated with these effects addition to conductivity for 2D-systems depends logarithmically on temperature. In Fig. 6 the dependence $\frac{\sigma(T)}{\sigma_0} - 1 = \frac{\Delta\sigma}{\sigma_0}$ vs. ln (T) is shown, where σ_0 is the conductivity at the temperature, for which the $\sigma(T)$ dependence deviates from the linear one. The linear dependence $\frac{\Delta\sigma}{\sigma_0}$ vs. ln (T) is observed for both the as-prepared and functionalized carbon nanotube, Fig. 6. Thereby, the obtained linear dependencies $\frac{\Delta\sigma}{\sigma_0}$ (ln (T)) indicate a possible manifestation of effects of weak localization and interaction of charge carriers for the two dimensional case in both carbon nanotube specimens.

Let us consider the obtained experimental data on the resistivity changes in magnetic field in the framework of the theories of weak localization and interactions of charge carriers for the two – dimensional case. Such approach was successfully used for the analysis of resistivity changes in the magnetic field and estimates of Fermi energy level for N-doped carbon nanotube [15].

The quantum effects addition to conductivity in the magnetic field can be written as

$$\Delta\sigma_q(B) = \Delta\sigma_{loc}(B) + \Delta\sigma_{el-el}(B), \qquad (1)$$

where $\Delta\sigma_{loc}(B)$ is the contribution to conductivity in magnetic field due to the weak localization, and $\Delta\sigma_{el-el}(B)$ the contribution to conductivity due to the interaction. The contribution to the conductivity due to the effect of charge carriers weak localization is determined as [19–21]:

$$\Delta\sigma_{loc}(B) = \frac{e^2}{2\pi^2\hbar} Y\left(\frac{4eDB\tau_\varphi}{\hbar}\right)$$
$$Y(x) = \begin{cases} \dfrac{x^2}{24} & \text{at} \quad x \ll 1 \\ \ln(x) & \text{at} \quad x \gg 1 \end{cases} \qquad (2)$$

where D is diffusion coefficient: $D = \frac{L_T^2 k_b T}{\hbar}$ (L_T is the coherence length in conductor) and τ_φ the phase relaxation time: $\tau_\varphi = A^* T^{-p}$ (A^* and p are coefficients). The characteristic field for weak localization effect is determined as $B_{loc} = \frac{\hbar}{4eD\tau_\varphi}$. Contribution to the two – dimensional conductivity in magnetic field due to interaction effects appears as:

$$\Delta\sigma_{el-el}(B) = \frac{e^2}{2\pi^2\hbar} G(T,B)\, f\left(\frac{2eDB}{\pi k_B T}\right)$$
$$f(x) = \begin{cases} 0.3x^2 & \text{at} \quad x \ll 1 \\ \ln(x) & \text{at} \quad x \gg 1 \end{cases} \qquad (3)$$

where G(T, B) is the parameter of charge carrier interaction. The characteristic field in this case is determined from the relation $B_{el-el} = \frac{\pi k_B T}{2eD}$. Thus, according to (2) and (3) additive to the total conduction in case of magnetic fields less characteristic is proportional to the square of the magnetic field, and the above characteristic field is proportional to the logarithm of the magnetic field. *Fig. 7* presents the experimental dependences of conductivity on magnetic field at low temperatures $\frac{\Delta\sigma(B)}{\sigma_0} = \frac{\sigma(B)}{\sigma_0} - 1$ vs. B for specimens of as-prepared and functionalized carbon nanotubes. The experimental dependence $\frac{\Delta\sigma(B)}{\sigma_0} = f(B)$ for both carbon nanotubes specimens can be approximated by square field dependence ($y_1 = a_1B^2$) at low magnetic fields and by logarithmic dependence ($y_2 = b + a_2 \ln(B)$) at high magnetic fields, Fig. 7. Analogous dependences are observed for both carbon nanotube spezimens and at higher temperatures up to 35 K. Thus, for both specimens of as-prepared and functionalized carbon nanotubes additives to conductivity in magnetic field are proportional to the square of magnetic field at low fields and to the logarithm of the magnetic field at high magnetic fields.

Let us estimate the value of characteristic field in case charge carriers interction. Taking into account the expression for the diffusion coefficient D: $B_{el-el} = \frac{\pi k_B T}{2eD} = \frac{\pi k_B T\hbar}{2eL_T^2 k_B T} = \frac{\pi\hbar}{2eL_T^2}$. Let us assume that in the first approximation the coherence length L_T equals to effective length of the charge carriers free path, which is determined by crystallites size in

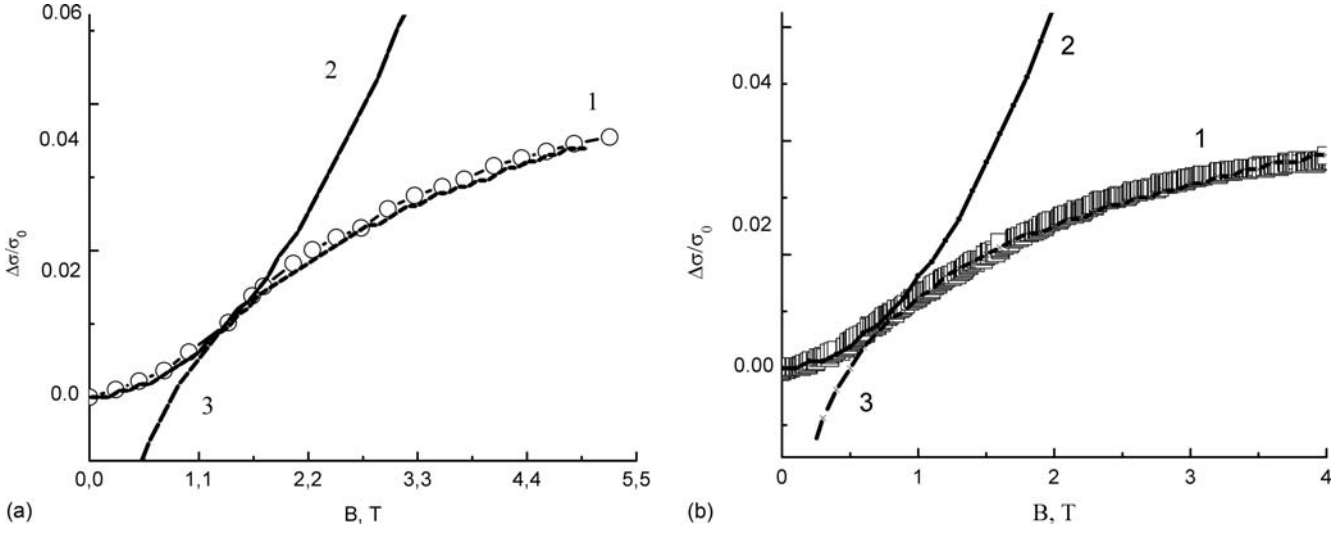

Figure 7. $\Delta\sigma(B)/\sigma_0$ dependence at low temperatures for specimens of as-prepared ((a), T = 4.2 K) and functionalized ((b), T = 1.6 K) carbon nanotubes, 1 – experimental data, 2 – approximation $y_1 = a_1B^2$, 3 – approximation $y_2 = b + a_2 \ln(B)$

carbon nanotube and don't dependend on temperature [15]. Thus B_{el-el} doesn't depend on temperature, and it is determined only by the sizes of crystallites in the investigated carbon nanotubes. The values of characteristic field B_{el-el} is amount to ~3.5 T for as-prepared carbon nanotube and ~5.9 T for functionalized carbon nanotube. For both carbon nanotubes specimens the magnetic fields, where a deviation from square dependence of $y_1 \sim a_1 B^2$ is observed, are significantly smaller, Fig. 7. Obviously, this is because the weak localization characteristic fields are significantly smaller than characteristic fields in case of charge carriers' interaction.

Let us define the temperature dependence of localization characteristic field B_{loc}. For this take the logarithm of the expression for localization characteristic field taking into account temperature dependences of diffusion coefficient D and phase relaxation time τ_φ:

$$\ln(B_{loc}) = \ln\left(\frac{\hbar^2}{4eL_T^2 k_b A^*}\right) + (p-1)\ln(T) \qquad (4)$$

The values of localization characteristic fields B_{loc} can be estimated from the experimental data in Fig. 7 as magnetic fields at which the dependence $\frac{\Delta\sigma(B)}{\sigma_0} = f(B)$ deviates from the squared one. *Fig 8* presents the determined temperature dependences of localization characteristic fields B_{loc} in the logarithmic coordinates. From these dependences the values p and A* for as-prepared and functionalized carbon nanotubes were estimated by using the Eq. (4). For as-prepared carbon nanotubes the value of p equals to 1.03 and temperature dependence of phase relaxation time $\tau_\varphi(T)$ can be written as $\tau_\varphi = 2.25 \cdot 10^{-12}\, T^{-1.03}$. Thus, for as-prepared carbon nanotube with a good approximation it is possible to consider temperature dependence of phase relaxation time $\tau_\varphi(T)$ as $\tau_\varphi = \frac{A^*}{T}$. This result agrees very good with temperature dependence of phase relaxation time $\tau_\varphi = A^* T^{-0.98}$ obtained for pure multiwall carbon nanotube in [15]. For functionalized carbon nanotubes p = 1.36, and $\tau_\varphi = 3.82 \cdot 10^{-12} T^{-1.36}$ that is the temperature dependence of phase relaxation time is more pronounced. Thus, from the experimental dependence of conductivity on magnetic field in the terns of 2D-weak localization, the temperature dependences of wave functions phase relaxation time were obtained for as-prepared and functionalized carbon nanotubes.

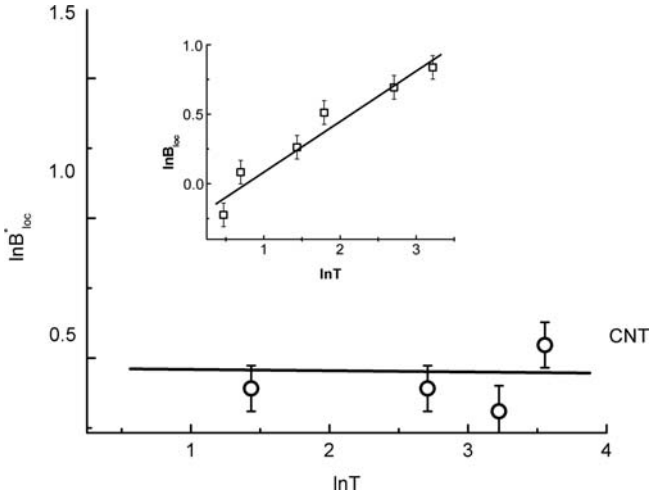

Figure 8. Temperature dependences of localization characteristic fields B_{loc} in the logarithmic coordinates for as-prepared and functionalized (inset) carbon nanotubes

Consider the contribution to the two-dimensional conductivity due to effects of charge carriers weak localization and interaction in the case of weak magnetic fields (magnetic field smaller, than the characteristic magnetic field). According to the theory of weak localization and interaction this contribution in such magnetic fields is proportional to the square of the magnetic induction:

$$\Delta\sigma_{B^2} = \frac{\sqrt{3}e^2 D^2 \gamma_0 a_0}{2\pi E_F L_T}\left(\frac{\tau_\varphi^2}{3\hbar^2} + \frac{1.2G}{\pi^2 k_B^2 T^2}\right)B^2 = a_1 B^2 \qquad (5)$$

Let us consider in details the coefficient a_1 at B^2 which we denote as a_{1CNT} for as-prepared carbon nanotubes and a_{1FCNT} for functionalized carbon nanotubes. In the expressions for the coefficients a_{1CNT} and a_{1FCNT} we can write in explicit form corresponding expressions for diffusion coefficient D and the relaxation time τ_φ. For as-prepared carbon nanotube the expression for a_{1CNT} takes the form:

$$a_{1CNT} = \frac{\sqrt{3}e^2 L_T^3 k_B^2 \gamma_0 a_0}{2\pi E_F \hbar^2}\left(\frac{(A^*)}{3\hbar^2} + \frac{1.2G}{\pi^2 k_B^2}\right) \qquad (6)$$

Therefore, for as-prepared carbon nanotube coefficient a_{1CNT} doesn't depend on temperature.

For functionalized carbon nanotubes dependence of a_{1FCNT} from temperature is complicated:

$$a_{1FCNT} = \frac{\sqrt{3}e^2L_T^3k_B^2\gamma_0 a_0}{2\pi E_F\hbar^2}\left(\frac{(A^*)^2}{3\hbar^2T^{0.72}} + \frac{1.2G}{\pi^2k_B^2}\right) = C_1T^{-0.72} + C_2 \quad (7)$$

where

$$C_1 = \frac{\sqrt{3}e^2L_T^3k_B^2\gamma_0 a_0(A^*)^2}{2\pi E_F\hbar^4}$$

$$C_2 = \frac{1.2\cdot\sqrt{3}e^2L_T^3k_B^2\gamma_0 a_0}{2\pi^3E_F\hbar^2k_B^2}$$

Thus, a_{1FCNT} is linear depend from $T^{-0.72}$.

Fig. 9 presents the temperature dependences of coefficients a_{1CNT} and a_{1FCNT} (inset). These coefficients were determined as coefficients at B^2 in dependences type $y = a_1B^2$. Experimental dependences $\frac{\Delta\sigma(B)}{\sigma_0} = f(B)$ at low magnetic field have been described with such dependence, Fig. 7. For as-prepared carbon nanotube an insignificant temperature dependence of coefficient a_{1CNT} is observed, Fig. 9. On the other hand the linear dependence of a_{1FCNT} vs. $T^{-0.72}$ can be clearly seen in Fig. 9 (inset). The values C_1 and C_2 were determined from the experimental dependence $a_{1FCNT}(T^{-0.72})$ and allowed to estimate the energy of Fermi level $E_{F\,FCNT}$ and the constant G, that are equal: $E_{F\,FCNT}$ = 0.054 eV, G = 0.22 (constant G is much less than 1 in case of small magnetic fields) [21]. Let us use the obtained value of constant G for determination of the Fermi level energy for as-prepared carbon nanotube: $E_{F\,CNT} = 0.083$ eV. Thus, according to our research the functionalization of carbon nanotube with the proposed method results in a slight decrease in the value of the Fermi level energy. This result differs significantly for instance from previously published results [15]. It has been shown that the doping of carbon nanotube with nitrogen causing a significant increase (almost 10 times) the value of the Fermi energy level. Let us analyze the possible reasons for these differences. It is well known that nitrogen doping of carbon nanotubes creates an electron donor state in the conduction band near the Fermi level and leads to growth of the density of delocalized charge carriers in multi walled carbon nanotube [22]. This is manifested, in particular, in reducing the value of resistivity. On the other hand the nitrogen doping of carbon nanotube doesn't significantly affect the structure of individual tube and doesn't change the surface condition of individual tubes. Most other processes occur at functionalization of the carbon nanotube by the proposed method. The proposed functionalization method, despite the fact that it uses such strong oxidants as sulfuric and nitric acids, also doesn't lead to the destruction of the carbon nanotube internal structure and the creation of significant defects that is confirmed by X-ray diffraction and electron microscopy. As shown by our study, such functionalization does not significantly affect the band structure and electronic spectrum of carbon nanotube. Such functionalization causes change of surface states of individual tubes by the addition of functional groups, which in turn leads to a significant increase in contact resistance between the individual functionalized tubes. Note that the contact resistance between as-prepared tubes in investigated specimens (high specimen's density, high concentration of tubes in specimens) is not significant in comparison with the same resistance tubes.

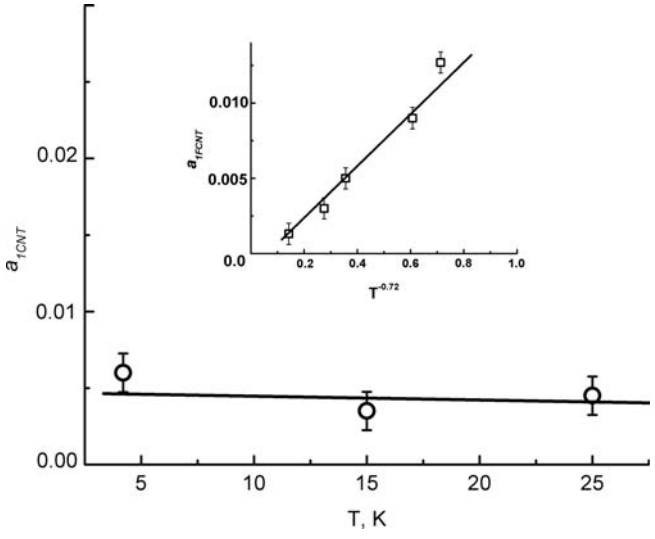

Figure 9. Temperature dependences of coefficients a_{1CNT} and a_{1FCNT} (inset)

The relative contribution to two-dimensional conductivity in case of strong magnetic fields that is $B > B_{el-el}$ takes the form:

$$\Lambda\sigma_{\ln B} = \frac{\sqrt{3}\gamma_0 a_0}{2\pi E_F L_T} \times \left(\ln\left(\frac{4eDB\tau_\varphi}{\hbar}\right) + G\ln\left(\frac{2eD}{\pi k_B T}\right) + (1+G)\ln(B) \right) \quad (8)$$

At a constant temperature the dependence (8) can be rewritten in the form: $\Delta\sigma_{\ln B} = b_2 + a_2(1+G)\ln(B)$. Logarithmic dependences of $\Delta\sigma_q$ from B are really observed experimentally at high magnetic fields, Fig. 7. However, it is rather difficult to obtain any quantitative estimates in this interval of magnetic fields because constant G depends complicated manner on temperature and magnetic field:

$$\frac{1}{G(B)} = \frac{1}{\lambda} + \ln\left(\frac{c\eta}{eBD}\right) \text{ and}$$

$$\frac{1}{G(T)} = \frac{1}{\lambda} + \ln\left(\frac{\gamma\eta}{\pi k_B T}\right).$$

4 Summary

The carried out investigations of magnetoresistance of as-prepared and fuctionalized tubes allowed estimating the influece of proposed functionalizations method on the electronic structure and charging transfer in carbon nanotube. Our investigations revealed that the proposed functionalization method doesn't cause any new defects in structure of carbon nanotubes. It is shown that functionalization of carbon nanotubes with proposed method doesn't essentially influence on temperature dependence of resistivity and only slightly increases the resistance value. Low-temperature investigations of resistivity and magnetoresistance revealed that for as-prepared and functionalized carbon nanotube the manifestation of the charge carriers weak localization and interactions effects took place. These effects lead to irregular temperature and field dependences of resistance, in particular, in the manifestation of a negative magnetoresistance. Based on the experimental data the explicit type of temperature dependence of the wave function phase relaxation time and Fermi-level energy for as-prepared and functionalized carbon nanotubes are established. In our opinion revealed increasing of resistivity of carbon nanotube under functionalization is caused by change of surface conditions under functionalizarion. Such change in surface conditions leads to increasing of contact resistance.

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