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Stability of the Tomonaga–Luttinger liquid state in gamma – irradiated carbon nanotube bundles

**B A Danilchenko¹, N A Tripachko¹, E A Voitsihovska¹, I I Yaskovets¹,
I Y Uvarova¹ and B Sundqvist²**

¹Institute of Physics NASU, Pr. Nauki 46, 03028 Kiev, Ukraine

²Department of Physics, Umeå University, SE-901 87 Umeå, Sweden

E-mail: danil@iop.kiev.ua

Abstract. We report experimental results for the changes in conductivity of single-wall carbon nanotube bundles when irradiated by ^{60}Co γ – rays in various environments. In the current study the samples investigated were irradiated in hermetic cells, either evacuated (0.1 Pa) or filled with hydrogen or deuterium at atmosphere pressure. *In situ* measurements of the resistance change as a function of irradiation dose at room temperature are presented. It was found that for all irradiation conditions, the normalized resistance versus irradiation dose demonstrates a logarithmic behaviour. A phenomenological model for the observed dependence is derived. The current–voltage characteristics of the irradiated samples were measured in the temperature range from 4.5 to 300 K using short (10 ns) electric pulses and the results demonstrate a scaling behaviour. This scaling occurs in the universal coordinates that correspond to the Tomonaga–Luttinger liquid concept. The results obtained confirm the existence of the Tomonaga–Luttinger liquid phase up to room temperature in carbon nanotubes after γ – irradiation to a dose of 5×10^7 rad in vacuum, 1.7×10^7 rad in hydrogen and 1.24×10^8 rad in deuterium.

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1. Introduction

Since their discovery, carbon nanotubes have been considered unique one-dimensional nanostructures with outstanding physical properties. Their atomic, electronic, transport, sorption and other properties suggest a variety of practical applications and require further investigation [1]. In particular, the

influence of ion, electron, gamma and other irradiation types on these properties is still little known, in spite of numerous studies [2–5]. Recent experiments show that γ –irradiation of multi-walled (MWCNT) and single-walled (SWCNT) carbon nanotubes modifies their functionality [6, 7], enhances their hydrogen storage capacity [8, 9, 10] and strongly affects the parameters of SWCNT based field-effect transistors [11].

Investigations of carrier transport in SWCNTs play a basic role in the development of novel devices based on the unique properties of such one-dimensional structures. Among these properties, carrier transport in single SWCNTs and its bundles due to the Tomonaga–Luttinger (T–L) liquid [12, 13] conduction mechanism remains the most interesting [14–18]. The presence of defects and adsorbed atoms leads to a random electric potential distribution along the SWCNT, resulting in a finite probability for back-scattering of the electrons. In this context, the question of stability of the T–L state in SWCNTs arises. This problem was considered theoretically in many works [19–23] whereas the number of relevant experimental studies is limited [24].

In this paper, we report experimental results for the change in conductivity of SWCNT bundles when irradiated with ^{60}Co γ rays in hydrogen and deuterium media as well as in vacuum. These irradiation conditions were chosen for several reasons: γ –irradiation introduces structural defects, mostly vacancies [25], and the efficiency of defect creation depends strongly on the surrounding gas medium during γ –irradiation [26]. Moreover, γ –irradiation up to a dose of $1.7 \times 10^7 \text{ rad}$ in hydrogen leads to the accumulation of atomic hydrogen inside SWCNTs, with a concentration of up to 3.5 at% [10].

In this experiment we may thus study and compare the influence on the conductivity of SWCNTs by both intrinsic defects produced under γ –irradiation in vacuum or deuterium and by a significant concentration of atomic hydrogen impurities .

2. Experimental details

The samples investigated consisted of compressed SWCNT powder containing bundles (CCVD, Cheap Tubes, USA) and having a metallic conductivity [16, 27]. The diameters of individual nanotubes in the bundles were determined by Raman spectroscopy (excitation wavelength – 514.5 nm) to be 1.02–1.05 nm and 1.68–1.73 nm [27]. The original powder was compacted at room temperature by applying an uniaxial pressure of 1 GPa. High-resolution electron microscopy images [16, 27] show that the structure of the samples can be described as a system of multiply connected nanotube bundles with high porosity. Sample geometry, details of its preparation and characterization as well as the pulse method for the current–voltage ($J - V$) characteristics measurements are described in Refs. [16, 27].

All samples had electrical contacts for *in situ* resistance measurements under irradiation in real time and were inserted into hermetically closed, demountable cells. In order to remove extrinsic gases, cells were evacuated to 0.1 Pa for 24 hours at room temperature. After this the cells were filled with gases (hydrogen or deuterium at atmosphere pressure) or remained evacuated. The experimental set-up for room temperature ^{60}Co γ –irradiation is schematically depicted in figure 1. It should be mentioned that the metal walls of the cells utilized are transparent to ^{60}Co γ –rays with energies 1.2 MeV such that such rays reach the sample without attenuation. Moreover, the γ –ray flux density is homogeneous and isotropic due to the specific geometry of the ^{60}Co gun.

3. Results and discussion of the resistance change caused by the radiation

The measured resistance change $\Delta R = [R(\phi) - R_0]$, normalized to the initial resistance at room temperature R_0 as a function of irradiation dose ϕ is presented in figure 2. For all investigated samples this dependence shows a non-monotonic behavior. The resistance initially decreases with irradiation dose and then rises at higher doses. The sample irradiated in vacuum shows an almost unnoticeable resistance decrease of down to -0.2% at a dose $\phi = 2 \times 10^7 \text{ rad}$ followed by an increase of up to 15% at a dose $\phi = 5.2 \times 10^7 \text{ rad}$. Irradiation in hydrogen demonstrates a weak linear decrease down to -1.7% at a dose $\phi = 8 \times 10^6 \text{ rad}$ and then increases by up to 11% at a dose of $3.5 \times 10^7 \text{ rad}$. The most pronounced normalized resistance slope is observed for the sample irradiated in deuterium where the resistance

decrease reaches -17.5% at the dose $\phi = 2.5 \times 10^7 \text{ rad}$. After that, the resistance rises gradually up to 17% at a dose $\phi = 1.24 \times 10^8 \text{ rad}$.

Such a behavior is the result of several mechanisms associated with the influence of irradiation on the conductivity of the nanotube bundles. A resistance decrease is usually associated with radiation induced links between nanotubes in the bundle [28] and also with electron wave function delocalization in the vicinity of radiation defects in nanotube walls [21]. At the same time, a resistance rise is related to nanotube amorphization [28] and electron scattering on damaged areas of individual nanotubes [29].

The elementary unit of radiation induced damage in the SWCNT shell is a single vacancy [2]. We have simulated several configurations of such a vacancy in a (4, 4) armchair SWCNT (figure 3) using density functional theory (DFT) and the software package Gaussian-03 [30] with B3LYP functional [31] and the 6-31 G* basis set. The calculated metastable (figure 3a) and stable (figure 3b) configurations are similar to configurations previously presented for (6, 6) armchair SWCNT [2]. According to our calculation the “butterfly”-like configuration (figure 3c) has the lowest energy.

Our experimental results demonstrate an asymptotic behavior in which a plot of $\Delta R / R$ versus ϕ approaches the logarithmic function $\Delta R / R_0 \propto \ln(\phi / \phi_0)$ with a free parameter ϕ_0 (see figure 2). Below, we show that the observed resistance behavior is an intrinsic property of an array of nanotube bundles or ropes.

The sample investigated can be represented as a random net of N_0 conducting channels formed by nanotubes. The total sample conductivity is defined as $G = G_0 \times N_0$, where G_0 is the conductivity of one channel. Under irradiation, the number of conducting channels is $N = N_0 - N^*$, where N^* is the number of channels with defects. For simplicity, we assumed that a nanotube with a single radiation damage loses its conductivity [29]. In the limit $N^* / N \ll 1$, the relative resistance change can be found as $\Delta R / R_0 \propto N^* / N_0$. The rate of generation of radiation damage can be written as

$$\frac{dN^*}{dt} = \lambda(1 - \nu) \quad (1)$$

where ν is the fraction of nanotubes with radiation damage. Here $\lambda = I \cdot \sigma \cdot M$, I is the intensity, σ is the cross-section for carbon atom displacement in a nanotube and M is the number of carbon atoms per unit volume.

The creation of dN^* nanotubes with radiation damage changes their fraction by the value $d\nu$. When $d\nu$ increases, the number of undamaged nanotubes decreases and becomes $(1 - \nu)$. Therefore, the differential equation for ν can be written as

$$\frac{d\nu}{dN^*} = V_0(1 - \nu) \quad (2)$$

with V_0 is a constant. Substituting the solution of (2) into (1) we have

$$N^* = \frac{1}{V_0} \ln(\lambda V_0 t + 1). \quad (3)$$

Taking into account that the irradiation dose is $I \times t = \phi$ and introducing $\sigma \times M \times V_0 = \phi_0^{-1}$, the relative resistance change is found to be

$$\frac{\Delta R}{R_0} \propto \frac{1}{N_0 V_0} \ln(\phi / \phi_0 + 1). \quad (4)$$

The results of the measurements in vacuum are well described by Eq. (4) (see figure 2). In contrast, irradiation in a hydrogen or deuterium atmosphere demonstrates significant effects due to the generation of inter-tube links that were not taken into account in the derivation above. If we restrict ourselves to the asymptotic dependence of the resistance at high doses, Eq. (4) can be written in a more general form as $\Delta R / R_0 \propto \ln(\phi / \phi_0 + \delta)$.

4. Experimental evidence of the Luttinger liquid conductivity and discussion

There are several theoretical models to describe the conductivity of carbon nanotubes arrays, which in our particular case consist of compacted nanotube bundles. The basic models are the mechanism of variable range hopping conductivity (VRH) [32, 33], the tunnel mechanism or fluctuation-induced tunneling (FIT) [34] and Luttinger liquid conductivity [12, 13, 35, 36].

The first two mechanisms are often used for the interpretation of experimental results for the temperature dependent conductivity in the low electric field limit [3, 4, 37]. Both theories predict that the conductivity depends exponentially on temperature or applied voltage and do not follow any scaling relations. In contrast, the Luttinger liquid conductivity theory [35, 36] predicts that the conductivity depends on temperature and applied voltage in such a way that it asymptotically approaches a power law. This theory introduces universal coordinates in which the current-voltage characteristics measured at different temperatures follow a scaling law. Therefore, such a scaling behavior of the carrier transport serves as experimental evidence for the validity of the T–L concept.

Samples were withdrawn from the cell for electrical measurements at the doses $0.47 \times 10^7 \text{ rad}$ and $1.7 \times 10^7 \text{ rad}$ (depicted in figure 2 by crosses, line II) for samples irradiated in hydrogen and at the doses $5.2 \times 10^7 \text{ rad}$ and $1.24 \times 10^8 \text{ rad}$ for samples irradiated in vacuum and deuterium, respectively. The resistance–temperature ($R - T$) measurements were carried out in the temperature range 4.5 – 330 K in direct current mode (DC) under a weak electric field. Normalized resistance values $R(T) / R_{300K}$ versus temperature T for the initial state and after γ -irradiation are shown in figure 4.

It is seen that the results of $R - T$ measurements for the sample irradiated in hydrogen up to $0.47 \times 10^7 \text{ rad}$, in vacuum up to $5.2 \times 10^7 \text{ rad}$ and in deuterium up to $1.24 \times 10^8 \text{ rad}$ almost coincide with the results obtained before irradiation. In the temperature interval 30 – 330 K the normalized resistances have the form of a power function $R/R_0 = 9.25 \times T^{-\alpha}$ with an exponent $\alpha = 0.4$. Below 30 K, the resistance deviates from this power function due to hopping conductivity. Such results are typical for SWCNT bundles with metallic conductivity [16, 27, 38].

After the irradiation in hydrogen up to the dose $1.7 \times 10^7 \text{ rad}$ the temperature dependence of the resistance differs significantly from the other curves (see figure 4), nevertheless, at temperatures above 200 K, this dependence also approaches the power function $R/R_0 = 9.25 \times T^{-\alpha}$ with the same α . For this sample the $J - V$ characteristics were investigated in detail. The measurements were performed by using short electric pulses of 10 ns duration (for details, see Refs. [16, 27]). This method allows us to avoid heating effects in the case of large electric fields. The $J - V$ characteristics were measured at the temperatures 5.5, 12, 50, 100, 175, 190, 200 and 300 K and the results are presented in figure 5, panel I.

Similar measurements were carried out for the sample irradiated in vacuum up to the dose $5.2 \times 10^7 \text{ rad}$ (depicted in figure 2 by crosses, line I). The results obtained at the temperatures 5.5, 12, 24, 50, 100, 190, 200 and 290 K are depicted in figure 6. The $J - V$ characteristics measured for the sample irradiated in deuterium to the dose $1.24 \times 10^8 \text{ rad}$ (depicted in figure 2 by crosses, line III) are shown in figure 7, panel I.

In the T–L model, the current through each SWCNT is described by the expression [35]

$$J = I_0 T^{\alpha+1} \sinh\left(\frac{eV_s}{2k_B T}\right) \times \left| \Gamma\left(1 + \frac{\alpha}{2} + i \frac{eV_s}{2\pi k_B T}\right) \right|^2 \quad (5)$$

where I_0 is a constant, e and k_B are the electron charge and the Boltzmann constant, respectively, $\Gamma(x)$ is the gamma function and V_s is the voltage drop over a single nanotube. The exponent α is a theoretical parameter that is determined by the electron-electron interaction in the nanotube. There are two asymptotes for Eq. (5): $V/J \propto R \propto T^{-\alpha}$ and $J \propto V^{1+\alpha}$ [35, 36] in the low and high field limits, respectively. Such a power law dependence is expected for the T–L liquid [12, 13] and arises from the peculiar dependence of the single-particles density of states $n(q) \propto (q - q_F)^\alpha$ on the wave vector q in the vicinity of the Fermi wave-vector q_F . In the T–L liquid model the value of the non-universal exponent α is determined by the strength of the electron-electron interaction in the nanotube itself.

In our study, the low field limit is reached as shown in figure 4 with $\alpha = 0.4$ in the temperature interval 30 – 330 K, with the exception of the sample irradiated in hydrogen to the dose

$1.7 \times 10^7 \text{ rad}$. Experimental results from $J - V$ pulse measurements at 12 and 5.5 K demonstrate the tendency toward the high field asymptotic power function behavior with $\alpha = 0.43 - 0.45$, see figure 5 – 7.

In the universal coordinates, $J / T^{1+\alpha}$ versus $eV_s / k_B T$, all characteristics should collapse to a single curve as long as the studied system remains in the T–L liquid state. Initial experimental data (shown in panels I in figures 5 – 7) demonstrate that experimental and calculated results converge to a single curve in these coordinates (figures 5 – 7 panels II).

We use a dimensionless parameter β allowing us to introduce a value $V_s = \beta \times V$ which is included in Eq. (5). Here V_s means the voltage drop on an elementary fragment of a nanotube. The effective number of such fragments between the electric contacts in the sample is $W = 1/\beta$, where W is the sample length. All the results from $J - V$ measurements in the temperature interval 5.5 – 300 K converge to a single curve at $\alpha = 0.45 - 0.5$. This single curve coincides with Eq. (5) with the fitting parameter $\beta = 7 \times 10^{-4}$ for the sample irradiated in hydrogen, with $\beta = 6.5 \times 10^{-4}$ for the sample irradiated in vacuum and $\beta = 5.3 \times 10^{-4}$ for the sample irradiated in deuterium. This means that we have experimental arguments for the applicability of the T–L liquid concept in the temperature interval 5.5 – 300 K, and that we can use this model to describe the current flow in the γ – irradiated SWCNT bundles.

5. Conclusions

We have performed an experimental study of the influence of ^{60}Co γ – ray irradiation on the electrical properties of SWCNT bundles with metallic conductivity. The SWCNT samples were irradiated at room temperature in hydrogen and deuterium media at atmosphere pressure as well as in vacuum. *In situ* measurements of the resistance changes demonstrate a logarithmic behavior as a function of irradiation dose. Also, a phenomenological approach for describing the observed dependence is proposed. According to this model such a logarithmic resistance behavior versus irradiation dose is an intrinsic property of the array of coupled conductive SWCNTs bundles that were investigated. The derived dependence $\Delta R/R_0 \propto \ln(\phi/\phi_0 + \delta)$ describes well the experimental data obtained for the sample irradiated in hydrogen up to $0.47 \times 10^7 \text{ rad}$, in vacuum up to $5.2 \times 10^7 \text{ rad}$ and in deuterium up to $1.24 \times 10^8 \text{ rad}$.

Irradiated SWCNT bundles demonstrate a scaling behavior of the measured current–voltage characteristics in the temperature interval 5.5 – 300 K. The scaling occurs in the universal coordinates that follow from the Tomonaga–Luttinger liquid concept. The measured $J - V$ characteristics converged to a single curve in the coordinates $J / T^{1+\alpha}$ versus $\beta \times eV / k_B T$ and coincided with the analytical Eq. (5) calculated using only one fitting parameter β . The curves coincide over a range of values of the dimensionless energy parameter $\beta \times eV / k_B T$ of six orders of magnitude, from 10^{-5} to 10^1 .

The results obtained demonstrate the existence of the Tomonaga–Luttinger liquid phase at room temperature after γ – irradiation up to a dose of $5 \times 10^7 \text{ rad}$ in vacuum, $1.7 \times 10^7 \text{ rad}$ in hydrogen and $1.24 \times 10^8 \text{ rad}$ in deuterium. Thus, the Tomonaga–Luttinger liquid demonstrates the stability of its parameters both under conditions of radiation induced defects and under extremely high concentrations (3.5 at%) of absorbed atomic hydrogen.

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Figure Captions:

Figure 1. Schematic view of the experimental set-up for the γ – ray irradiation of a SWCNT sample in the hermetic cell, either under vacuum, in hydrogen, or in deuterium. The sample geometry and size are only shown schematically. Conductivity measurements were carried out *in situ* under irradiation.

Figure 2. Normalized resistance change as a function of irradiation dose ϕ for different irradiation conditions: Line I – irradiation in vacuum, line II – in hydrogen, line III – in deuterium. Crosses indicate the doses after which $R - T$ and $J - V$ measurements were carried out. Solid lines show the function $\Delta R/R_0 \propto \ln(\phi/\phi_0 + \delta)$, with $\phi_0 = 3.9 \times 10^7 \text{ rad}$ and $\delta = 1$ for irradiation in vacuum, $\phi_0 = 2 \times 10^7 \text{ rad}$ and $\delta = 0.45$ for irradiation in hydrogen, $\phi_0 = 5.5 \times 10^7 \text{ rad}$ and $\delta = 0.01$ for irradiation in deuterium.

Figure 3. Calculated configurations of a single vacancy in a (4, 4) armchair SWCNT. Carbon atoms on the front shell of the SWCNT near the vacancy are shadowed for better visualization. Reconstruction of a single vacancy: *a* – metastable, *b* – stable, *c* – “butterfly”-like configuration with the lowest energy.

Figure 4. Temperature dependence of the normalized resistance of SWCNT bundles at different irradiation doses. Symbols denote: initial state (+), irradiated in hydrogen with the doses $0.47 \times 10^7 \text{ rad}$ (●) and $1.7 \times 10^7 \text{ rad}$ (■), irradiated in vacuum with the dose $5.2 \times 10^7 \text{ rad}$ (□) and in deuterium with the dose $1.24 \times 10^8 \text{ rad}$ (○). The solid line is a power function $R \propto T^{-\alpha}$ with $\alpha = 0.4$.

Figure 5. Panel I: $J - V$ characteristics at several temperatures for carbon nanotubes irradiated in hydrogen. From the bottom upwards the curves show data obtained at 5.5, 12, 50, 100, 175, 190 and 300 K (curves are not labeled to keep the figure legible). Panel II: the measured $J - V$ characteristics plotted in the universal coordinates $J / T^{1+\alpha}$ versus dimensionless energy parameter $\beta \times eV / k_B T$, with $\beta = 7 \times 10^{-4}$. The dashed line is an asymptotic power law function $J \propto V^{1+\alpha}$ with $\alpha = 0.45$. Solid line is the result of a calculation with Eq.(5).

Figure 6. Panel I: $J - V$ characteristics of carbon nanotubes irradiated in vacuum measured at several temperatures. From the bottom upwards the curves show data obtained at 5.5, 12, 24, 50, 100, 190, 200 and 290 K (curves are not labeled to keep the figure legible). Panel II: the measured $J - V$ characteristics plotted in the universal coordinates $J / T^{1+0.45}$ versus $\beta \times eV / k_B T$, with $\beta = 6.5 \times 10^{-4}$. Solid line is the result of a calculation with Eq.(5).

Figure 7. Panel I: $J - V$ characteristics of carbon nanotubes irradiated in deuterium, measured at several temperatures. From the bottom upwards the curves show data obtained at 6, 12, 24, 50, 165, 210 and 290 K (curves are not labeled to keep the figure legible). Panel II: the measured $J - V$ characteristics plotted in the universal coordinates $J / T^{1+0.5}$ versus $\beta \times eV / k_B T$, with $\beta = 5.3 \times 10^{-4}$. Solid line is the result of a calculation with Eq.(5).

FIGURES:

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7