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Original article

Conducting properties of single-wall carbon nanotubes in composites based on polystyrene

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ABSTRACT Composite films were synthesized by radical copolymerization of styrene with methacrylate groups on the surface of modified single-walled carbon nanotubes. Mechanical grinding and reforming of films on the electrode led to a decrease in the electrical resistance values by two magnitude orders. This effect was observed when measuring the current-voltage characteristics in both sandwich and planar structures. This decrease in the electrical resistance of the composite films is likely due to the disintegration and reorientation of carbon nanotubes, as well as the creation of mechanical stresses in them as a result of covalent bonding to the polymer matrix, which could affect the electronic structure of carbon inclusions.

KEYWORDS single-walled carbon nanotubes, surface modification, polymer composites, electrical resistance

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1. Introduction

Research concerning the electrical conductivity of single-walled carbon nanotubes (SWCNTs) was intensively carried out in recent decades, including for their use as fillers in polymer composites for flexible electronics and other applications [1–5]. It is known that the conductivity values for SWCNTs can vary from dielectric and semiconducting to metallic, depending on the method of their preparation, axial ratio and surface modification [6–11]. The introduction of disaggregated SWCNTs in quantities sufficient for percolation, even into high-resistivity polymers, made it possible to obtain composites with high electrical conductivity values, up to 57 S/cm [12]. Yoon H. and colleagues [13] investigated which method of dispersing millimeter-long SWCNTs was the most effective for obtaining composites with high electrical conductivity characteristics based on fluorinated rubber and polystyrene (PS). The authors used a total of 11 approaches to disperse SWCNTs in methyl isobutyl ketone, which were based on three mechanisms of action: turbulent flow (nanomizer, high-pressure jet mill), cavitation (probe sonicator) and mechanical forces (ball-mill, bead-mill, paint shaker, ball collision-mill, cone-mill, high shear batch disperser, thin-film spin mixer, rotor-mill). The comparative analysis of dispersion methods presented in [13] showed that the highest average electrical conductivity of SWCNTs in a fluorine rubber film was 33 S/cm and was achieved by grinding the filler in a turbulent solvent flow, preceding it mixing with the polymer, molding and drying of the composite film. In turn, ultrasonic and mechanical effects on the dispersion of SWCNTs in methyl isobutyl ketone made it possible to obtain polymer composites with lower electrical conductivity of 20 and 8 S/cm, respectively. For polystyrene-based samples, the trend of changes in the electrical conductivity of SWCNTs depending on the method of their dispersion remained unchanged [13]. Thus, an increase in the electrical conductivity of the composites was achieved due to the homogeneous distribution of SWCNTs during their dispersion in a turbulent flow, which allowed one to form a branched network of conducting channels of large range with a minimum number of breaks in the polymer matrix. In work [14], in order to avoid severe destruction of SWCNTs under the influence of ultrasound, the authors resorted to their mechanical grinding and found that this led to an increase in the electrical conductivity of nanotubes in films where ethyl cellulose acts as a binder. The electrical conductivity of the composite films depended on SWCNTs grinding duration (5, 15, 60 min) and their concentration relative to the binder (1:1, 1:10, 1:50). An increase in the grinding time of SWCNTs from 15 to 60 minutes had a negative effect on the electrical conductivity of the composite films, which was associated, in the authors opinion, with excessive aeration of the filler during the crushing process. A clear explanation for the higher electrical conductivity of ethylcellulose films with SWCNTs subjected to five-minute grinding in comparison with similar compositions based on uncrushed carbon filler was not given in this work. Another study [15] was devoted to comparing theoretical and experimental data on the electrical conductivity of SWCNTs with different lengths and axial ratios. Theoretical calculations showed that longer SWCNTs in polymers should have better conductivity, while the experimentally obtained electrical conductivity values for short and long nanotubes were in the same range ($10^4 \div 10^8$ S/m).

Uniform distribution and a decrease in the aggregation degree of SWCNTs can be achieved not only by dispersion, but also by using their surface functionalization. To do this, during the oxidation process, the SWCNT framework was damaged by strong acids, oxidizing agents, ozone or plasma, which resulted in the generation of oxygen-containing functional groups, such as hydroxyl, carboxyl and ester [16, 17]. These groups were subsequently used for silanization [18], esterification [19] of SWCNTs and grafting of polymers onto them [20]. It should be noted that in works [8,21,22], the authors discovered growth in the number of charge carriers and an increase in the electrical conductivity of SWCNTs during their oxidation. Moreover, some articles [23, 24] talk about the manifestation of the superconductivity effect in SWCNTs, which can also make a significant contribution to reducing the electrical resistance of the composite material. Similar effects was previously observed for another allotropic modification of carbon, namely reduced graphene oxide (rGO), when its sheets were deformed and mechanical stresses were created in them during copolymerization with styrene, that led to the appearance of local areas with abnormally low electrical resistance and the manifestation of superconducting properties in the samples up to room temperatures [25–27]. In the present work, we study the influence of the SWCNT's surface functionalization process with methacrylate groups, their subsequent copolymerization with styrene, precipitation conditions, disintegration and formation of a polymer composite coating on the conductive properties of 1D carbon filler.

2. Experimental

The studied single-walled carbon nanotubes (obtained from OCSiAl) according to the manufacturer have a length of more than 5 μm, an outer average diameter of 1.4 ± 0.3 nm and a specific surface area of about 400 m^2/g. SWCNTs taken in their original form were functionalized with methacrylate groups using the organosilicon compound 3-(trimethoxysilyl)propyl methacrylate (TMSPM) (Sigma-Aldrich, CAS number 2530-85-0, purity >98 %) according to the method described in detail in [28]. After removing unreacted TMSPM from the alcohol dispersion of modified SWCNTs by repeated washing with ethanol and drying the resulting powder in an air to constant weight, the fact of grafting of the organosilicon modifier to the carbon filler was confirmed using FTIR spectroscopy (Vertex 70 spectrometer, Bruker, Germany). The transmission spectrum of a KBr tablet with original SWCNTs (Fig. 1a) contained barely noticeable bands at 1730 (stretching vibrations of C=O groups), 1510 (skeletal vibrations of C=C bonds) and 1250 cm^{-1} (stretching vibrations of C–O groups, corresponding to internal defects of carbon nanotubes) [29, 30]. After modification of SWCNTs using TMSPM, bands appear in the FTIR spectrum (Fig. 1b) at 1720 (stretching vibrations of C=O groups), 1634 (C=C groups), 1270 (Si–CH_3 bonding), 1190 (Si–O–CH_3 vibrations), 1096 (Si–O–Si antisymmetric stretching) cm^{-1}, that indicates the presence of an organosilicon modifier on the surface of the carbon filler [31, 32]. Another indirect confirmation of the successful modification of the surface of carbon nanotubes was an increase in their diameter after treatment with TMSPM. It should be noted that the smallest diameter of original SWCNTs recorded using a SUPRA 55 VP scanning electron microscope (Carl Zeiss, Germany) was about 4 nm (Fig. 2a), while after their functionalization it approximately doubled (Fig. 2b).

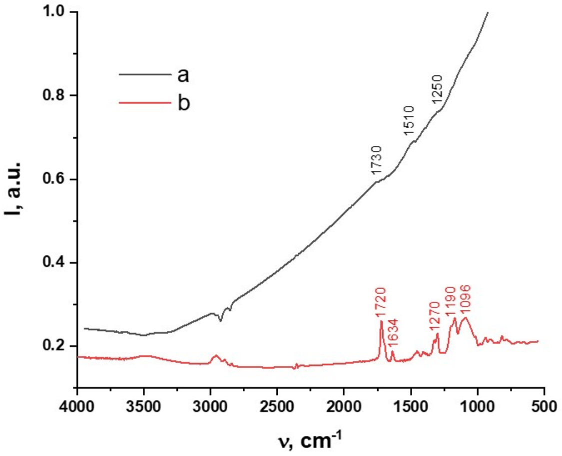
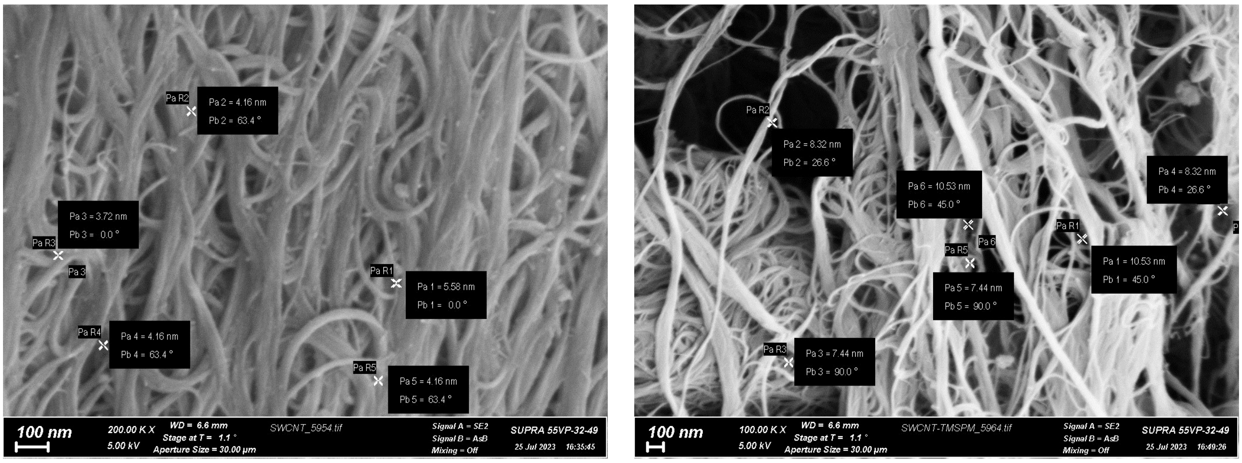


FIG. 1. FTIR spectra of SWCNTs before (a) and after (b) treatment with TMSPM

Next, the functionalized SWCNTs were dispersed in a styrene–toluene solution (1:1) and copolymerized in an inert atmosphere with the monomer for 20 hours, using azobisisobutyronitrile (1 wt. % of the polymer weight) as an initiator. The content of SWCNTs in the synthesized composite was 1 wt. %. The average molecular weight of polystyrene under the selected polymerization conditions was about 9000 Da. The resulting composite was precipitated in ethanol and, after



(a) (b)

FIG. 2. SEM micrographs of original (a) and TMSPM-modified (b) SWCNTs

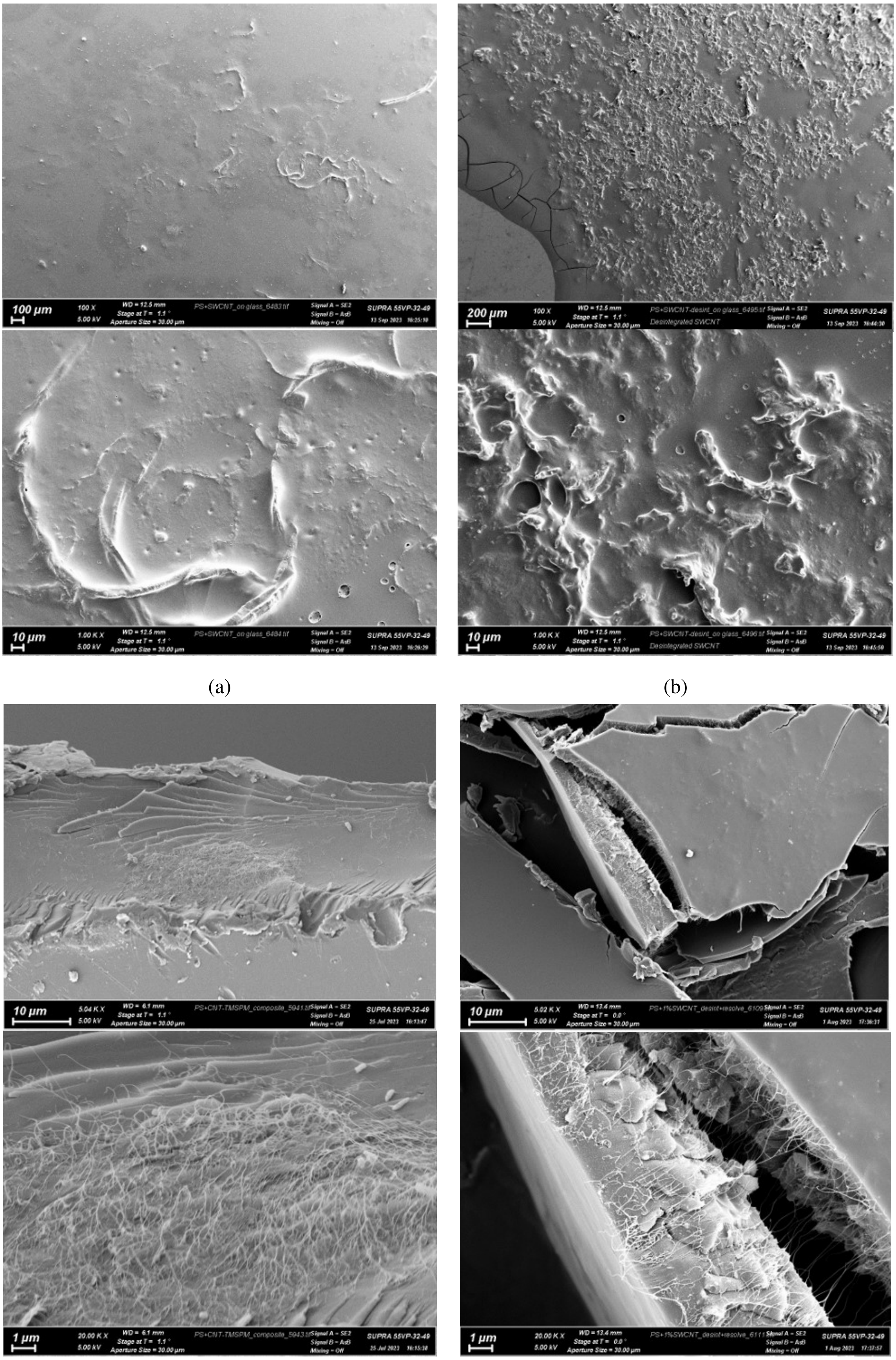
drying, divided into 2 parts. One part was left unchanged (original), the other part was mechanically crushed in a vibrator ball mill (DDR-GM 9458, 30 W, 50 Hz, Germany) for 2 hours to a powdery state.

The current-voltage characteristics of the composites were measured in two ways: 1) in the geometry of the sandwich structure; 2) in the planar geometry. To obtain current-voltage characteristics (CVCs) in metal/composite/metal sandwich structures, the samples were deposited onto copper electrodes from their 1 % solutions in benzene. The area of the copper electrodes used to measure electrical conductivity of the composites in sandwich structures was 1 cm^2. The thickness of the films deposited on the electrodes for CVCs measuring was from 2 to 10 μm, and the temperature dependencies of the electrical resistance were obtained for the samples of 2 and 3 μm. The thickness of the composite coating was estimated using a Solver P-47 PRO (NT-MDT) scanning probe microscope. CVCs and temperature dependencies of the electrical resistance in metal electrode/composite/metal electrode sandwich structures were obtained by 4-probe method as in [25, 33]. The probe contacts were soldered into the current electrodes. In this case, the voltage drops across the electrodes at the maximum current used in the experiment was less than the sensitivity of the voltmeter, i.e. 100 nV. The following devices were used in electrical measurements: universal voltmeter V7-78/1, multimeter RS-232C, combined digital device Shch-300, DC power supply B5-90.

To measure CVCs in planar structures, the composites were deposited on glass substrate from a mixture of benzene/petroleum ether solvents taken in a ratio of 1:1 by volume. To prepare solutions, chemical pure benzene (chemical pure, ECOS-1, Russia) and petroleum ether 40 – 70 (chemical pure, ECOS-1, Russia) were used. Previously, it was shown [33] that this ratio allowed the maximum separation of rGO and polystyrene and promoted the maximum enlargement of carbon clusters on the polymer surface. To obtain sufficiently large SWCNT structures on the polystyrene surface deposited from a mixture of benzene and petroleum ether, solutions were prepared with a composite content of about 30 wt. %. For the film obtained from a non-disintegrated composite, the length of SWCNT formations emerging on the polystyrene surface reached 500 μm (Fig. 3a). At the same time, on the surface of the film formed on a glass substrate from the composite crushed to a powder state, SWCNT aggregates with a size of 20 – 30 μm were observed (Fig. 3b). Such sizes of inclusions made it possible to measure their electrical resistance on the surface of the polymer using the copper electrodes with an area of 10 μm^2.

3. Results and discussion

Since PS is a dielectric, it is an ideal matrix for studying the electrical conductivity of a component with lower resistance, such as SWCNTs. Composite coatings of uncrushed and disintegrated PS films with SWCNTs formed on a glass substrate using a mixed solvent were heterogeneous in their electroconductive properties. Low electrical resistance was observed only in some small (up to hundreds of micrometers) areas of composites, while most of their surface remained high-resisted or has electroconductivity corresponding to semiconductors. When the electrodes were separated by more than 1 cm, the electrical resistance of the composite film exceeded 30 MΩ, since the low concentration of SWCNTs (1 wt. %) in the PS did not allow the formation of extended electroconductive channels in the near-surface layer of the dielectric matrix. The metallic type of conductivity was recorded only for individual areas of the composite film (Fig. 4), where the SWCNTs came out onto the PS surface. The length of such areas corresponded to the sizes of the agglomerates observed in the SEM micrographs of cross-sections of the composite films (Fig. 3c). It is obvious that the structural features of the aggregates and the relative arrangement of the SWCNTs in the PS film formed using a mixed solvent of benzene/petroleum ether determine the electrical resistance values of its areas. Low values of electrical



(a) (b)

(c) (d)

FIG. 3. SEM micrographs obtained at different magnifications of the surface of the films formed from the original (a, c) and disintegrated (b, d) composites on the glass substrate (a, b), as well as images of their transverse cleavages (c, d)

resistance at the distance between electrodes, down to 50 μm, were recorded in planar structures both for films formed from uncrushed composite and those obtained from the pre-disintegrated sample (Fig. 4). It is shown that the absolute values of electrical resistance of agglomerates from disintegrated SWCNTs in the PS matrix can be even lower than that of metal. It would be logical to expect better electrical conductivity in the case of the composite films with uncrushed SWCNTs, when their structure was not damaged and there were fewer edge defects, which should obviously appear when the nanotubes were disintegrated. However, in the case of disintegrated SWCNTs, the electrical resistance in planar structures was 2 orders of magnitude lower than that of the composite films based on uncrushed ones. Apparently, the reduction in the electrical resistance of the films was facilitated by the special self-organization of individual SWCNT fragments into aggregates that acted as conducting channels in the dielectric matrix.

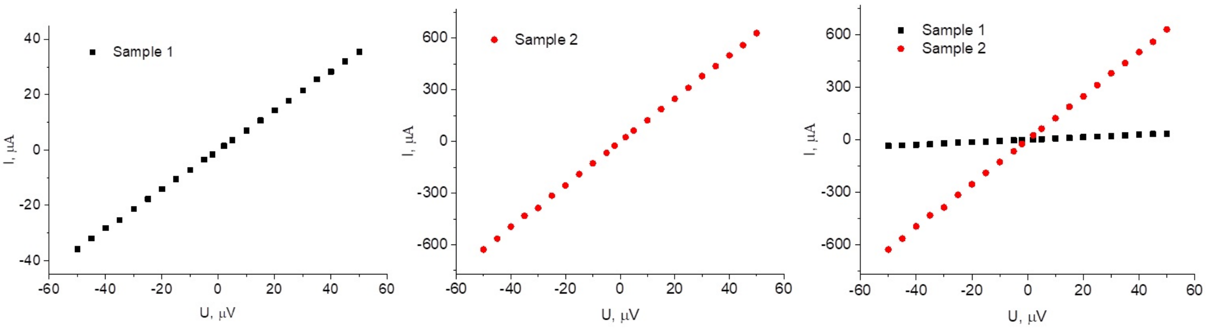


FIG. 4. CVCs at 298 K in planar-structures of composite films based on PS with original (sample 1, $R = 1.4\ \Omega$) and disintegrated SWCNTs (sample 2, $R = 0.08\ \Omega$); distance between electrodes 50 μm

To measure the electrical conductivity of the composites in sandwich structures, the films with a thickness from 2 to 10 μm were formed on the copper electrodes from benzene. In this case, SWCNTs covalently bonded to PS were uniformly distributed and did not form large aggregates in the polymer matrix (Fig. 3b). The electrically conductive properties of the composite films were determined by their thickness depending on the length of the SWCNTs. The films obtained from the pre-disintegrated PS samples with SWCNTs showed low electrical resistance values at a thickness of up to several micrometers (Fig. 5), and over 5 μm they became completely insulating. This can be explained by the fact that during grinding, the length of some part of individual SWCNTs was shortened, and they ceased to provide electrical conductivity at a film thickness much greater than their linear dimensions. Thus, for disintegrated SWCNTs in sandwich structures, low electrical resistance was maintained only at small film thicknesses, comparable to the sizes of the conducting channels they form (Fig. 3d). For the films based on PS with uncrushed SWCNTs, the electrical conductivity was maintained even at a thickness of 10 μm. However, if we compare thin films obtained from uncrushed and disintegrated composites, the absolute values of electrical conductivity for the latter will be significantly higher (comparison of sample 1 and 2 in Fig. 5).

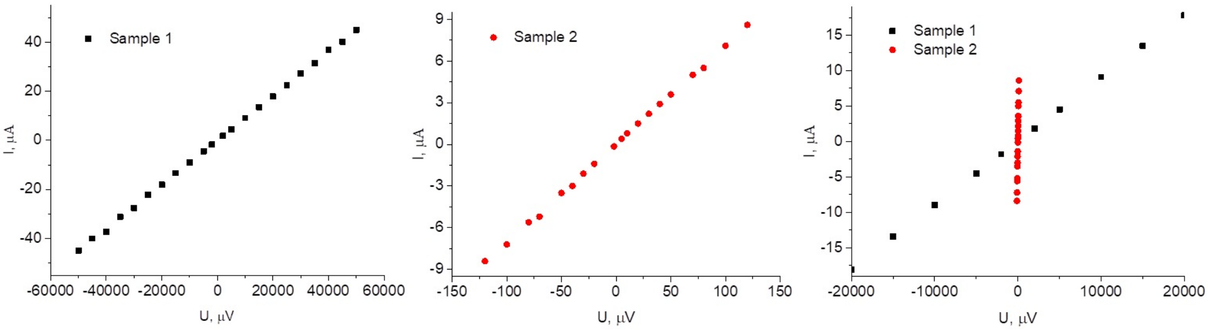


FIG. 5. CVCs at 298 K in sandwich-structures of composite films based on PS with original (sample 1, $R = 1.4$ kΩ) and disintegrated SWCNTs (sample 2, $R = 14\ \Omega$); distance between the electrodes 2 μm

The metallic nature of the electrical conductivity of SWCNTs in PS films formed from the original and disintegrated parts of the polymer composite was also confirmed by the temperature dependencies of the resistance (Fig. 6). Thus, at 298 K the electrical resistance of the SWCNTs was 220 kΩ, at 280 K it was already 650 kΩ, and at 273 K it was 1 MΩ. In addition, the electrical resistance of the films obtained from the disintegrated PS composites with SWCNTs dropped by approximately 100 times when the temperature was reduced from room to liquid nitrogen temperature (Fig. 6, sample 2). For the samples 2 and 3 at low temperatures, electrical resistance of copper level was observed in the experiment.

Therefore, for clarity, a comparative analysis of the temperature dependences of the resistance of the PS samples with the original and disintegrated SWCNTs was carried out with a film thickness of 3 μm (Fig. 6).

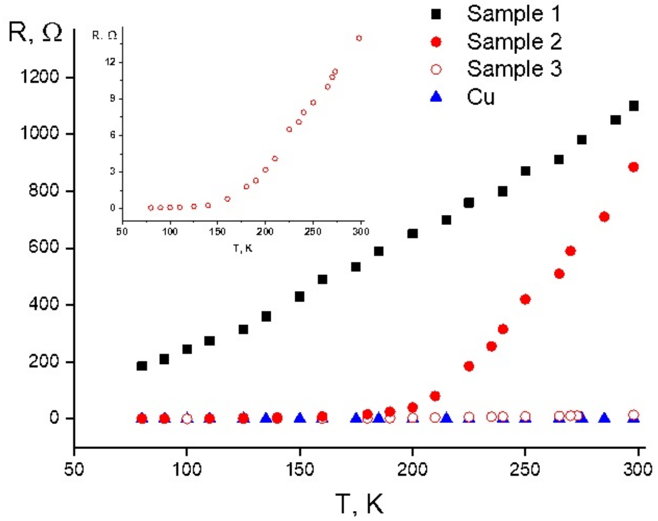


FIG. 6. Temperature dependencies of the electrical resistance of the composite films based on PS with original (sample 1, film thickness 3 μm) and disintegrated SWCNTs (sample 2, film thickness 3 μm; sample 3, film thickness 2 μm) measured in planar structures in comparison with copper

4. Conclusions

For the films deposited from solutions of original and disintegrated polymer composite obtained by radical polymerization of styrene with SWCNTs, the current-voltage characteristics and the temperature dependencies of the resistance have been measured. The obtained data have demonstrated a decrease in resistance and a metallic type of conductivity in certain areas of the nanotube aggregates' surface. It has been determined that the grinding of SWCNTs leads to a decrease in their resistance in planar structures in separate areas by two orders of magnitude compared to original ones. Also, when using thin films of the composite with a thickness of up to 2 μm in sandwich structures, the electrical resistance values of the disintegrated polymer composite turned out to be lower by two orders of magnitude comparing to original composite.

Thus, we can conclude that mechanical grinding of the composite material leads to the fragmentation of SWCNTs, and, consequently, to a change in the arrangement of the carbon filler in the network of conducting channels. We believe that, to varying degrees, both of these factors may contribute to the decrease in a local conductivity of the composite material based on polystyrene and SWCNTs observed in this work.

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Conflict of interest: the authors declare no conflict of interest.