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Citation for published version (APA):

Yu, J., Lu, K., Sourty, E., Grossiord, N., Koning, C. E., & Loos, J. (2007). Characterization of conductive multiwall carbon nanotube/polystyrene composites prepared by latex technology. *Carbon*, 45(15), 2897-2903. <https://doi.org/10.1016/j.carbon.2007.10.005>

DOI:

[10.1016/j.carbon.2007.10.005](https://doi.org/10.1016/j.carbon.2007.10.005)

Document status and date:

Published: 01/01/2007

Document Version:

Publisher's PDF, also known as Version of Record (includes final page, issue and volume numbers)

Please check the document version of this publication:

- A submitted manuscript is the version of the article upon submission and before peer-review. There can be important differences between the submitted version and the official published version of record. People interested in the research are advised to contact the author for the final version of the publication, or visit the DOI to the publisher's website.
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Characterization of conductive multiwall carbon nanotube/polystyrene composites prepared by latex technology

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Received 5 April 2007; accepted 8 October 2007

Available online 15 October 2007

Abstract

Conductive multiwall carbon nanotube/polystyrene (MWCNT/PS) composites are prepared based on latex technology. MWCNTs are first dispersed in aqueous solution of sodium dodecyl sulfate (SDS) driven by sonication and then mixed with different amounts of PS latex. From these mixtures MWCNT/PS composites were prepared by freeze-drying and compression molding. The dispersion of MWCNTs in aqueous SDS solution and in the PS matrix is monitored by UV-vis, transmission electron microscopy, electron tomography and scanning electron microscopy. When applying adequate preparation conditions, MWCNTs are well dispersed and homogeneously incorporated in the PS matrix. The percolation threshold for conduction is about 1.5 wt% of MWCNTs in the composites, and a maximum conductivity of about 1 S m^{-1} can be achieved. The approach presented can be adapted to other MWCNT/polymer latex systems.

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1. Introduction

Carbon nanotubes (CNTs) have attracted a great deal of interest from all over the world since the study published by Iijima in 1991 [1]. CNTs possess unique mechanical, thermal and electrical properties [2–5], such as a high elastic modulus (which can be larger than 1 TPa), thermal stability up to 2800 °C in a vacuum, thermal conductivity approximately twice as high as that of diamond, and so on. Their unique properties and high aspect ratio make CNTs a preferred candidate to be used as fillers in manufacturing high-performance and conductive composites.

Preliminary work in this field has shown some promising results [6–12]. Sandler et al. found that a very low single wall carbon nanotube (SWCNT) content of about 0.04 wt% made epoxy resins conductive [6]. As for enhancement of mechanical properties, Gorga and Cohen [10] mixed 1 wt% of multiwall carbon nanotubes (MWCNTs) into poly(methyl methacrylate) (PMMA) and obtained 170% improvement in its toughness.

One bottleneck for using CNTs as fillers in polymer-based composites is that as-produced CNTs invariably exist as aligned aggregates or bundles that are tightly bound by an estimated interaction of 500 eV/μm of tube length for SWCNT [13,14] or highly entangled in case of MWCNT. Therefore, the dispersion and incorporation of CNTs as individuals in polymer matrices is one of the most important and challenging tasks toward maximizing the translation of CNT properties to the composites.

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To incorporate CNTs in polymer matrices, several methods have been proposed, including melt-processing [15–17] and extrusion [18], mechanical stretching [19], spin coating [20], the use of latex technology [9,21] or magnetic fields [22], a coagulation method [23] and in situ polymerization [24,25]. In recent studies, others and we have demonstrated that latex technology successfully can be applied to prepare SWCNT/polymer composites. A very low percolation threshold of 0.3 wt% SWCNTs in PS matrix [21] and even lower percolation threshold of about 0.04 wt% SWCNTs in poly(vinyl acetate) (PVAc) [26] have been reported. For MWCNT, a very similar latex-based process to disperse MWCNTs into a polymer matrix has been described earlier by Dufresne et al. and the percolation threshold of about 3 wt% is reported [27]. However, investigation on the effect of the surfactant on the dispersion of MWCNTs and the ultimate conductivity of the composites, as well as monitoring the incorporating of MWCNTs in the highly viscous polymer matrix has not yet been performed systematically.

In the present study, we investigate the dispersion capability of MWCNTs in aqueous sodium dodecyl sulfate (SDS) solution and in the PS matrix by using UV–vis spectroscopy, transmission electron microscopy (TEM), electron tomography, and by scanning electron microscopy (SEM), respectively. The morphological results obtained are correlated with the conductivity of the composites.

2. Experimental

2.1. Materials

The MWCNTs used were purified thin MWCNTs (batch number Nanocyl®-3100) provided by Nanocyl Belgium. The surfactant used for the dispersion of the MWCNTs was SDS (90%) provided by Merck Chemical Co. The PS latex used was synthesized in our laboratory from aqueous styrene/SDS emulsion, and contains 27.9 wt% solids with an average molecular weight of about 1.5×10^6 g mol⁻¹. All dispersion experiments were carried out with distilled water.

2.2. Preparation of the MWCNT/SDS dispersion

The dispersion was prepared by mixing 0.02 g MWCNTs with 20 mL of an aqueous SDS solution in a flask and then sonicating the resulting mixture for different time under mild conditions. SDS/MWCNT ratios range from 1:10 to 4:1 by weight. All sonication processes were carried out with a horn sonicator (Sonic Vibracell VC750) with a cylindrical tip (10 mm end cap diameter). The output power was fixed at 20 W, thus delivering energy of 1100–1200 J/min. The flask is placed in a bath of ice water during sonication in order to prevent rising of the temperature of the mixture.

2.3. MWCNT/PS film processing

MWCNTs (1 wt%) were dispersed in 1.5 wt% SDS solution driven by sonication. After the maximum dispersion has been achieved, the dispersion was centrifuged at 3500 rpm for 30 min, at a Varifuge RF ES3000 with a Sepatech rotor (11 cm), to remove big MWCNTs bundles and catalyst particles. The supernatant was mixed with PS latex, the mixture was then frozen in liquid nitrogen for several minutes and the aqueous solvent was removed with a Christ Alpha 2–4 dryer operated at 0.2 mbar and

20 °C overnight. The resulting composite powder was compressed into films at 180 °C between poly(ethylene terephthalate) sheets with a Collin Press 300G.

2.4. Preparation of MWCNT buckypaper

Dispersions of 0.01 wt% MWCNTs in aqueous solution with different concentration of SDS were prepared by sonication. After the maximum dispersion was reached, 20 mL dispersion was poured into a filtration unit, and pressurized to push the suspension through a filter. MWCNTs deposited on the filter surface formed the MWCNT buckypaper after subsequent drying. Neat MWCNTs dispersions without SDS were prepared accordingly, however, because these dispersions are not stable for longer than few minutes, inspecting the degree of dispersion was performed only by eyes, and deposition of the dispersion was done fast.

2.5. UV–vis spectroscopic measurements

UV–vis absorption spectra were recorded with a Hewlett–Packard 8453 spectrometer operating between 200 and 1100 nm. Small sample volumes were taken regularly during the sonicating process (about 100 µl each, thus the sonicated volume under investigation is almost unchanged during the whole experiment) and diluted by 150 times, resulting in certain MWCNT contents that were suitable for UV–vis measurements. The blank used was the original SDS solution diluted by 150 times, under the same conditions as the samples themselves.

2.6. Transmission electron microscopy investigations

TEM observations of MWCNT after dispersing in aqueous solutions were performed using a Tecnai 20 (FEI Co.) operated at 200 kV. The samples were prepared by dipping a copper TEM grid in the MWCNT dispersion and subsequent drying. Additional TEM imaging of cross-sectional cut composite samples was performed. The as-prepared composite samples were sectioned at room temperature using an ultra-microtome (Reichert–Jung Ultracut E).

TEM electron tomography was performed with a TITAN-Krios (FEI Co.) operated in bright-field mode at 300 kV. General information on TEM-tomography can be found e.g. in the recent review from Weyland and Midgley [28]. In detail, the specimens were mounted on a Fischione tomography holder that allows high maximum tilt angles: commonly tilt-series were acquired at $\pm 70^\circ$ using the Saxton scheme with tilt steps of between 1.5° (at 0° tilt) and 0.5° (at 70° tilt). All tilt-series were obtained in a fully automated fashion using Xplore3D (FEI Co.) software. The 2D-projections were aligned, the tilt-axis was determined, and the data series was reconstructed by using Inspect3D (FEI Co.). Visualization of the 3D data series was performed with Amira (Mercury Computer Systems).

2.7. Scanning electron microscopy investigations

MWCNT/PS composite films were observed by using a XL30 ESEM (FEI Co.) equipped with a field emission electron source. High vacuum conditions were applied and a secondary electron detector was used for image acquisition. No additional sample treatment such as surface etching or coating with a conductive layer has been applied before surface scanning. Standard acquisition conditions for charge contrast imaging were used [29].

2.8. Electrical conductivity measurements

The electrical conductivity was measured using a standard four-point method. Parallel contact lines in 1 cm length and with 1 cm interval were drawn with colloidal graphite on the composite film or the buckypaper, and all conductivity measurements were performed at room temperature

with a Keithley 6512 programmable electrometer. For each sample, conductivity data represent the average value of 10 consecutive measurements.

3. Results and discussion

3.1. Overview on preparation of MWCNT/PS composites

Application of latex technology is a relatively new approach to incorporate CNTs inside any kind of highly viscous polymers, which can be synthesized by emulsion polymerization such as polystyrene (PS), PMMA and polyethylene [30,31], or which can be brought into artificial latex (e.g. by applying high shear conditions) [26]. Fig. 1 shows the schematic description of the multi-step process for making CNT/polymer composites by using latex technology. First, CNTs are dispersed in aqueous SDS solution driven by sonication. After the maximum dispersion has been achieved, the CNT dispersion is centrifuged to remove some catalyst particles and possible remaining CNT aggregates. For high purity CNT batches, about 10% or less of the initial material is lost during this step and remains in the residue of the centrifugation (as determined by thermal gravimetric analysis (TGA) measurements). Then, the supernatant is mixed with polymer latex, to form finally, after freeze-drying and subsequent compression molding, a composite film. For CNT concentrations above the percolation threshold the composites consist of homogeneously distributed CNTs forming a conductive network

in the polymer matrix. Following the above illustrated process of CNT/latex composites preparation, the resulting products of the present study are shown in Fig. 2: the stable MWCNT dispersion, the PS latex emulsion, and the final composite.

3.2. Exfoliation dynamics of MWCNTs in aqueous SDS solution

The first step in applying latex technology is the dispersion of MWCNTs in aqueous SDS solution, which can be monitored by UV–vis spectroscopy [32,33]. Fig. 3 shows the evolution of the maximum absorbance as a function of the total energy supplied to the solution, which describes the dispersion dynamics of MWCNTs in aqueous solutions with different concentrations of SDS. The general trends of the UV absorbance versus total energy curves obtained for all SDS/MWCNT solutions are very similar, except for very low SDS/MWCNT ratios. After an increase at the beginning of the sonication process, the value of maximum absorbance reaches a plateau value after a certain amount of supplied energy, which corresponds to the maximum achievable degree of dispersion of the MWCNTs in the aqueous SDS solutions. With higher SDS concentration, the plateau value of maximum absorption increases, the dispersion rate of MWCNTs is higher at the beginning of the sonication process, and the total energy required achieving the maximum dispersion of MWCNTs decreases.

Fig. 1. Schematic description of the multi-step process for preparation of CNT/polymer composites by using latex technology.

Fig. 2. Images of products resulting from the preparation process as described above: (from left) SDS/MWCNT mixture in water, MWCNT dispersion, PS latex emulsion, composite sheet.

Fig. 3. Evolution of the maximum absorbance as a function of the energy delivered to the solution for 0.1 wt% MWCNT in aqueous solution of different SDS concentration (0.1 wt% MWCNT and the indicated SDS concentrations are the start concentrations of the experiments, which will decrease slightly during the experiments because of sample taken for the UV–vis spectroscopy measurements). The sonicated solutions are diluted by a factor of 150 when taking UV–vis measurements.

It indicates that the SDS molecules can exert an “unzipping force” to disperse MWCNTs from bundles, which is supported by the high local shear provided by sonication.

The evolution of the dispersion of 0.1 wt% MWCNTs in solution proceeds similar for SDS concentrations of 0.15 wt% or higher, indicating that the minimum ratio of SDS to MWCNTs for optimum dispersion is about 1.5:1 by weight. The maximum dispersion of MWCNTs in solution cannot be achieved for lower SDS/MWCNTs ratios because sufficient surfactant is required to produce most probably an efficient coating on the surface of the MWCNTs to prevent re-aggregation [34].

In order to visualize the dispersion state of MWCNTs in solution and to support the interpretation of the UV–vis results, corresponding TEM investigations have been performed. Fig. 4 shows TEM bright-field images of MWCNTs, which were prepared from an aqueous SDS solution that had reached the maximum UV–vis absorption, indicating highest degree of dispersion (the weight ratio of SDS to MWCNTs is 1.5:1). Hardly any big nano-

tube aggregates can be seen in the TEM image (Fig. 4a). From the high resolution TEM image of Fig. 4b, it can be discerned that the MWCNTs used have an outside diameters of about 20 nm, and the MWCNTs are coated with a layer of SDS with the thickness of a few nanometers. Most probably, this layer is not a monolayer of SDS surfactant. No further details can be observed on the internal organization of the surfactant layer. Cryo-TEM investigations might provide more information about SDS/MWCNT interactions in solution, but are out of the scope of the present study.

During the sonication process, SDS molecules can help to disperse MWCNTs from bundles and prevent re-aggregation of MWCNTs by electrostatic repulsion [34]. On the other hand, the SDS molecules adsorbed on the MWCNTs surface are influencing the electrical conductivity of the system. Fig. 5 shows the effect of SDS/MWCNTs ratio on the conductivity of so-called buckypaper [35] consisting of dried SDS/MWCNT films. After introducing SDS the conductivity of these films decreases significantly from about $3 \times 10^3 \text{ S m}^{-1}$ for neat MWCNTs to about $1 \times 10^3 \text{ S m}^{-1}$ for a SDS/MWCNT ratio of 1.5 to even lower conductivity values of about $4.5 \times 10^2 \text{ S m}^{-1}$ for a ratio of 10. The SDS adsorbs on the MWCNT surfaces and partly blocks off charge transport through the MWCNTs network; hopping of charge carriers from one to the other MWCNT becomes more difficult. This results in significant decrease of the conductivity of the system. However, at least a SDS/MWCNT ratio of 1.5 has to be chosen to guarantee optimum dispersion of the MWCNTs in the aqueous SDS solution; but higher SDS/MWCNTs ratios should be avoided.

3.3. Electrical conductivity of MWCNT/PS composites

Fig. 6 shows the effect of MWCNT concentration on the volume conductivity of MWCNT/PS composites. A drastic increase in the electrical conductivity is obtained for the composites when the MWCNTs content attains about 1.5 wt%, indicating that the percolation threshold for formation of a conductive MWCNT network in the

Fig. 4. TEM images of (a) utmost dispersion of MWCNTs and (b) SDS-coated individual MWCNT prepared from 0.15 wt% SDS solution–0.1 wt% MWCNTs. The solution has been diluted 10 times before casting.

Fig. 5. Evolution of the conductivity of MWCNT/SDS films as a function of the weight ratio of SDS to MWCNTs in aqueous solution.

Fig. 6. Volume conductivity of MWCNT/PS composites as a function of MWCNT concentration. Values represent an average of 10 measurements; standard variation is below 10%.

PS matrix is reached. The conductivity of pure PS is in the order of $10^{-10} \text{ S m}^{-1}$, and for a MWCNT concentration of 1.9 wt% conductivity of 0.1 S m^{-1} can be achieved. For higher MWCNT concentrations no pronounced increase of the conductivity could be observed; however, the maximum conductivity of about 1 S m^{-1} has been obtained for composites with MWCNT concentration of 5.5 wt%.

3.4. Incorporation of MWCNTs in the PS matrix

To gain more information on the organization of MWCNTs in the polymer matrix we have applied a microscopy technique recently described in the literature [29], which is based on charge contrast imaging using a SEM. Fig. 7a shows a SEM image of the surface of a MWCNT/PS composite film with a MWCNT concentration of 2 wt%. The MWCNTs are represented by the bright lines. Because of the different capability for charge transport of the conductive MWCNT and the insulating polymer matrix, the secondary electron yield is enriched at the location of the MWCNT, which results in the contrast between the MWCNT network and the polymer matrix. The MWCNTs are homogeneously distributed in the polymer matrix; no MWCNT large aggregations are visible.

Because local charging of the polymer matrix around the MWCNTs may have rendered the average diameter of the MWCNTs to be larger than expected, which might be an indication for the presence of small MWCNT bundles, additional TEM investigations have been performed. The TEM bright-field image of a cross-sectional cut sample with a MWCNT concentration of 2 wt% is presented in Fig. 7b. The MWCNTs are represented by the hollow tubes having good contrast to the PS matrix; the dark edges of the MWCNTs are the multiple graphite layers. The TEM image indicates that most of the MWCNTs are individually dispersed and well incorporated in PS matrix. On the other hand, the fact that the 2D image represents a projection of the 3D volume of the cross-sectional cut sample, which has a thickness of about 100 nm, may cause that separated MWCNTs seem to overlap in the projection.

Inspecting the volume distribution of the MWCNTs within the PS matrix by applying electron tomography, we are able to state that the MWCNTs are not aggregated; most of them just lay close to each others without contacting each others (Fig. 7c, a video showing the full volume reconstructed is provided as [Supplementary material](#)). However, electron tomography cannot provide any information on the conductive network formed in the polymer matrix, because it is limited to sample thicknesses of the cross-section (about 100–200 nm); the length of the MWCNTs, however, is several micrometers so that the typical length scale for junctions within the network is much

Fig. 7. (a) SEM charge contrast image of the surface, (b) TEM image of the cross-section of MWCNT/PS composite film, and (c) volume reconstruction of the MWCNT distribution within the PS matrix. The MWCNTs concentration is 2 wt%.

larger than the sample thickness for low concentrations of MWCNT.

4. Conclusions

Composite films of MWCNT/PS are prepared by using latex technology. MWCNTs are first dispersed in SDS solution driven by sonication. SDS stabilizes and prevents re-aggregation of MWCNTs after disentangling and separation from bundles; however, it also affects the conductivity of the system and reduces charge transport at MWCNT junctions within the conductive network. Therefore the minimum SDS/MWCNT weight ratio of about 1.5/1 should be employed when preparing MWCNTs dispersions: for this ratio maximum dispersion of MWCNTs in aqueous SDS solution is reached and electrical conductivity reduction is limited. After centrifuging, mixing with PS latex, freeze-drying and subsequent compression molding, homogeneously dispersed MWCNTs are found in the PS matrix of the composites. A pronounced percolation threshold has been observed for a quite low value of 1.5 wt% MWCNTs in the PS matrix. A conductive MWCNT network is formed in the PS matrix, allowing the dramatically increase of volume conductivity from $10^{-10} \text{ S m}^{-1}$ for the neat PS to 1 S m^{-1} for the conductive composites.

Acknowledgments

The authors are thankful for the financial support of China Scholarship Council. Ir. N.M.B. Smeets is kindly acknowledged for preparing PS latex. The work forms part of the Dutch Polymer Institute (DPI) program on Engineering Polymers. The TEM investigations were performed at the Soft Matter Cryo-TEM Research Unit, Department of Chemical Engineering and Chemistry, Eindhoven University of Technology.

Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at [doi:10.1016/j.carbon.2007.10.005](https://doi.org/10.1016/j.carbon.2007.10.005).

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