

Preparation, Morphology, and Properties of Conducting Polyaniline-Grafted Multiwalled Carbon Nanotubes/Epoxy Composites

Jun Xu,^{1,2,3} Pei Yao,^{1,2} Zhongyi Jiang,¹ Huijun Liu,^{1,2,4} Xuan Li,² Litao Liu,² Mei Li,³ Yingzhe Zheng³

¹School of Chemical Engineering and Technology, Tianjin University, Tianjin 300072, People's Republic of China

²Center for Analysis, Tianjin University, Tianjin 300072, People's Republic of China

³College of Science, Civil Aviation University of China, Tianjin 300300, People's Republic of China

⁴Faculty of Engineering, University of Wollongong, New South Wales 2522, Australia

Received 4 March 2011; accepted 22 September 2011

DOI 10.1002/app.35677

Published online 17 January 2012 in Wiley Online Library (wileyonlinelibrary.com).

ABSTRACT: Polyaniline-grafted multiwalled carbon nanotubes (PANI-g-MWNTs)/epoxy composites were prepared by solution blending and mould casting. Transmission electron microscope, scanning electron microscope, differential scanning calorimetry, thermogravimetric analysis, electrical conductivity measurement, and tensile and flexural measurements were used to characterize the morphology, thermal, electrical, and mechanical properties of the composites. The results showed that MWNTs were encapsulated by conducting dodecyl benzene sulfonic acid-doped PANI forming a core (MWNTs)-shell (PANI) nanostructure. PANI coatings swelled in tetrahydrofuran and MWNTs were homogeneously dispersed in epoxy matrix. With PANI-g-MWNTs introducing into epoxy resin, curing reaction was promoted and thermal stability of the composites was enhanced. Because of conducting

PANI chains wrapping on the surface of MWNTs and well dispersion of MWNTs in epoxy matrix, electrical conductivity at room temperature of the composites was increased by seven orders of magnitude compared with neat epoxy. In the PANI-g-MWNTs/epoxy composites, terminal amino groups of PANI coatings reacted with epoxy matrix during curing reactions, which guaranteed interfacial adhesion between MWNTs and epoxy resin. Mechanical properties including tensile strength, Young's modulus, flexural strength, and flexural modulus of the composites were increased by 61%, 43%, 78%, and 49% compared with neat epoxy, respectively. © 2012 Wiley Periodicals, Inc. *J Appl Polym Sci* 125: E334–E341, 2012

Key words: carbon nanotube; conducting polymers; composites; morphology; properties

INTRODUCTION

Carbon nanotubes (CNTs) with excellent thermal, electrical, and magnetic properties have received much interest in fabricating advanced materials. Because of low density, high aspect ratio, extraordinary strength, and flexibility, CNTs seem to become ideal reinforcing fibers to fabricate composites.¹ Epoxy resins are used in electronic, paints, printed circuit

boards, and packaging materials and are established as thermosetting matrices of advanced composites. Incorporation of CNTs into epoxy holds the promise of delivering superior composites with enhanced thermal, electrical, and mechanical properties.^{2,3}

However, aggregation, entanglement, and poor interfacial adhesion with polymers limit CNTs used as reinforcements in composites.⁴ A number of methods have been used to improve dispersibility of CNTs in polymer matrices, such as sonication, using of surfactants, high shear mixing, and functionalization of CNTs by chemical modification.^{5–7} Attachment of functional groups and polymer chains to CNTs can increase solubility of CNTs in solvents, improve dispersion of CNTs in matrix, and enhance binding strength at polymer–CNTs interface.⁸

Realization of CNTs reinforcing epoxy can be achieved by improving dispersion of CNTs in matrix and guarantee interfacial adhesion between CNTs and epoxy.⁹ Among numerous efforts made to fabricate CNTs/epoxy composites, amino-functionalization of multiwalled carbon nanotubes (MWNTs) is in favor of improving interfacial adhesion in the composites.¹⁰

Correspondence to: P. Yao (pyaotjueducn@163.com).

Contract grant sponsor: Fundamental Research Funds for Central Universities of Civil Aviation University of China; contract grant number: ZXH 2009D008.

Contract grant sponsor: Tianjin Natural Science Foundation; contract grant number: 08JCYBJC09800.

Contract grant sponsor: Scientific Research Project of Civil Aviation University of China; contract grant number: CAUC2009ZD0202.

Contract grant sponsor: Scientists Serving Enterprise Action of Ministry of Science and Technology of China; contract grant number: 2009GJA10036.

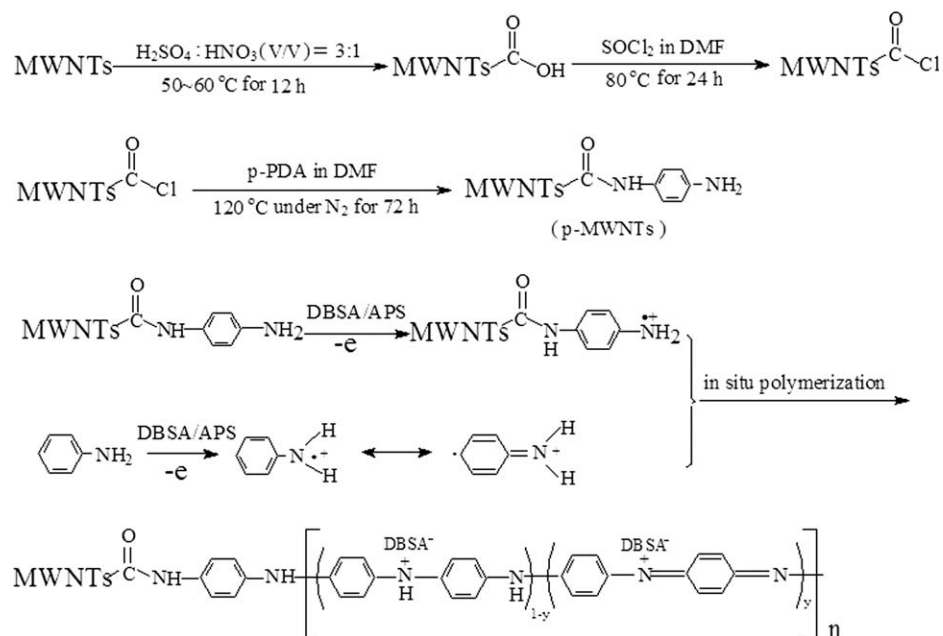


Figure 1 Chemical procedures for PANI-g-MWNTs nanostructure.

The terminal amino groups covalently attached to CNTs react with epoxy matrix; covalent bonds formed between functionalized CNTs and epoxy matrix provide interfacial shear stress and improve thermal and mechanical properties of epoxy.^{11,12}

It is known that CNTs have numerous π -bonds (C=C bond), and charges are transferred through the π -bonds of each CNTs.^{13,14} Recent studies reported that after CNTs were functionalized by nonconductive molecules, CNTs were isolated by insulated coatings, and the wrapped molecules perturbed π electron system (π - π conjugated electron system) of the CNTs walls.¹⁵ As a result, the composites exhibited only a marginal improvement in electrical conductivity, which was far inferior to that of unmodified CNTs/epoxy composite for a given amount of CNTs added.^{4,16}

Polyaniline (PANI) is a conducting polymer and received special attention because of its good processibility, environmental stability, and electrical properties by doping and protonation. Aniline *in situ* polymerization on the surface of MWNTs can prepare core (MWNTs)-shell (PANI) nanostructure, and the core-shell nanostructure is a good approach for dispersion of MWNTs in polymer matrix.¹⁷ Also, after monomers of PANI are introduced on the surface of MWNTs by covalent bond, they will join in polymerization, and therefore, conducting PANI chains are grafted on the surface of MWNTs. Combination of conducting PANI with MWNTs can tailor their properties and fabricate MWNTs-reinforced nanocomposites for electronic and mechanical applications.¹⁸ In our previous study,^{19,20} we introduced phenylamine groups ($-\text{C}_6\text{H}_4-\text{NH}_2$) on the surface

of MWNTs and synthesized organic soluble and conducting PANI-grafted multiwalled carbon nanotube core-shell nanostructure by *in situ* emulsion polymerization. The attachment of soluble DBSA-doped PANI chains on the surface of MWNTs via covalent bonding rendered MWNTs stable and well dispersed in tetrahydrofuran (THF).

In this article, we report a new epoxy composite reinforced by conducting PANI-grafted MWNTs (PANI-g-MWNTs) core-shell nanostructure. When PANI-g-MWNTs were dispersed in THF, PANI coatings swelled in THF and MWNTs were homogeneously dispersed in epoxy matrix. After evaporation of THF, addition of curing agent and curing reactions, PANI-g-MWNTs/epoxy composites were obtained. In the composites, MWNTs were wrapped by conducting PANI coatings in which π electron may transfer through MWNTs walls and conjugated PANI chains without perturbation. Also, PANI chains linked with MWNTs by covalent bond, terminal amino groups of PANI coatings reacted with epoxy matrix during curing reactions. These provided interfacial adhesion for load transfer in the composites. Chemical procedures for PANI-g-MWNTs nanostructure and sketch map of PANI-g-MWNTs reactions with epoxy are shown in Figure 1 and Figure 2, respectively.

EXPERIMENTAL

Materials

MWNTs (diameter = 20–30 nm, length = 5–15 nm) were provided from Chengdu Organic Chemicals Co., Ltd., Chinese Academy of Science R&D Center

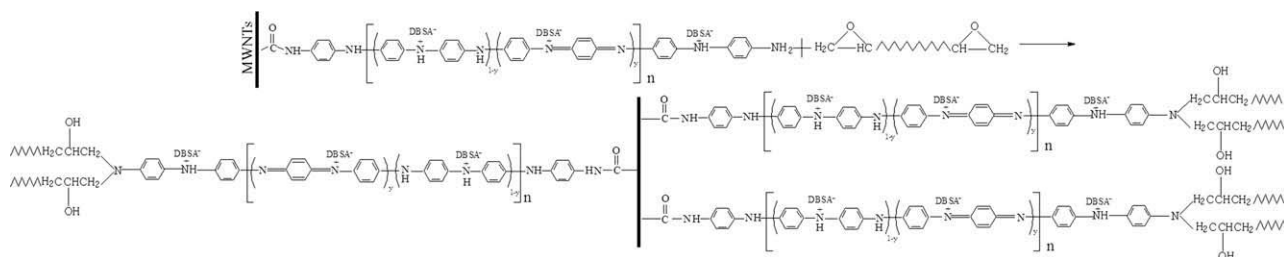


Figure 2 Sketch map of chemical reactions between PANI-g-MWNTs nanostructure and epoxy.

of Carbon Nanotubes, Chengdu, China. Preparation of phenylamine groups contained MWNTs (p-MWNTs) and DBSA-doped PANI-g-MWNTs core-shell nanostructure were according to our previous study.^{19,20} Purified MWNTs were ultrasonically treated with a 3 : 1 mixture of concentrated H_2SO_4 : HNO_3 at 50–60°C for 12 h and then dispersed in excess SOCl_2 and *N,N*-dimethyl formamide and refluxed at 80°C for 24 h converting carboxylic acid groups into acylchloride groups. After SOCl_2 was removed by distillation, excess *p*-phenylenediamine (*p*-PDA) was added in the mixture stirring at 120°C for 72 h in nitrogen atmosphere. Resulting p-MWNTs were filtered, washed and dried under vacuum. p-MWNTs were dispersed in DBSA aqueous solution by mechanical stirring and ultrasonication for 1 h, then aniline (mass ratio of p-MWNTs to aniline = 1 : 10) was added dropwise to p-MWNTs/DBSA micellar dispersion. After micellar dispersion was stirred and ultrasonicated for 2 h, ammonium peroxydisulfate (APS) aqueous solution was dripped into reaction system for 0.5 h (molar ratio of DBSA to aniline to APS was 0.8 : 1 : 1). Whole synthetic process was controlled at 30°C with constant mechanical stirring and ultrasonication. After polymerization was continued for 6 h, reaction was terminated by pouring acetone into the emulsion systems. Precipitate was filtered and washed with acetone, ethanol, and distilled water. The products (DBSA-doped PANI-g-MWNTs) were dried under vacuum at 60°C for 12 h. Tetrahydrofuran (analytical grade) was purchased from Tianjin Chemicals Co., Ltd. (Tianjin, China). Epoxy resin (DGEBA type, E-44) was purchased from Tianjin Yanhai Chemical Co. Ltd., (Tianjin, China). Curing agent (2-ethyl-4-methylimidazole, EMI-2,4) was purchased from Tianjin Yuanli Chemical Co. Ltd. (Tianjin, China).

Preparation of PANI-g-MWNTs/epoxy composites

Appropriate amount of PANI-g-MWNTs was weighted according to the required percentage and was dispersed in THF by mechanical stirring and ultrasonication for 1 h at 35°C to form a homogeneous suspension. Then epoxy resin (E-44) was added with mechanical stirring and ultrasonication for next

1 h at 45°C. After evaporation of THF, curing agent (EMI-2,4) was stirred into PANI-g-MWNTs/epoxy resin blending system at 45°C for another 0.5 h, weight ratio of curing agent to epoxy resin is 7 : 100. After that, the mixture was placed in a vacuum chamber to eliminate entrapped air and remaining THF at 50°C for 1 h. At last, the mixture was poured into Teflon mould and cured at 80°C for 2 h and 120°C for 2 h to form samples for characterization and measurements. The weight percentage of PANI-g-MWNTs in the composite samples were 0.25, 0.50, 0.75, 1.0, 1.25, and 1.50 wt %, respectively. For example, 1.081 parts of PANI-g-MWNTs by weight, 100 parts of epoxy resin, and 7 parts of curing agent by weight were used for preparing the PANI-g-MWNTs/epoxy composite sample with 1.0 wt % ($1.081/(1.081 + 100 + 7) = 1.0$ wt %) PANI-g-MWNTs content, and density of the cured composite sample was 1.189 g cm^{-3} . For comparative study, neat epoxy cured by EMI-2,4 was prepared under the same conditions without PANI-g-MWNTs addition.

Characterization and instrumental analysis

X-ray photoelectron spectroscopy (XPS) measurement of PANI-g-MWNTs nanostructure was carried out with a ESCA PHI-1600 PE XPS spectrometer with Mg ($K\alpha$) X-ray source. Core-level spectrum was referenced to C1s neutral carbon peak at 284.6 eV and was deconvoluted into Gaussian component peaks. Curve fitting was done by PHI Multipack 8.0 software. Morphology of p-MWNTs, PANI-g-MWNTs core-shell nanostructure, and PANI-g-MWNTs/epoxy resin blending system were carried out with an FEI TECNAI G2-F20 field emission transmission electron microscope (TEM) operated at 200 kV; fracture morphology of neat epoxy and PANI-g-MWNTs/epoxy composite were recorded under a PHILIPS XL-30 environmental scanning electron microscope (SEM) operated at 20 kV after samples were sputtered with gold. Initial curing temperatures (T_i), exothermic peak temperatures (T_p), and finish curing temperatures (T_f) of neat epoxy and the composite curing by EMI-2,4 were determined on a Perkin Elmer Pyris VII differential

scanning calorimetry (DSC) under nitrogen atmosphere; samples were heated from 40 to 200°C at a heating rate of 5°C min⁻¹. Thermal stability of neat epoxy and the composites were conducted with a TA SDTQ600 TG/DTA thermogravimetric analysis (TGA) system at a heating rate of 10°C min⁻¹ from room temperature up to 600°C under nitrogen gas atmosphere. Electrical conductivities of neat epoxy and the composites were measured at room temperature based on standard four-probe methods using specimens with dimensions 10 × 10 × 1 mm³ on a programmable SDY-5 voltage/current detector (Guangzhou Semi-Conductor Institute, Guangzhou, China). Tensile and flexural properties of neat epoxy and the composites were measured on a Testometric M350-50 kN universal testing machine at room temperature following ASTM-D638 and ASTM-D790, respectively. Dimensions of specimens were 120 × 13 × 3 mm³, and crosshead speed was 5 mm min⁻¹. The reported values are averages obtained with five specimens.

RESULTS AND DISCUSSION

XPS analysis

XPS spectra and deconvolution of N1s for PANI-g-MWNTs nanostructure are shown in Figure 3. The atomic concentrations of C1s, O1s, N1s, and S2p are 81.1, 12.3, 4.3, and 2.3%, respectively. It is found from Figure 3(b) that N1s of PANI-g-MWNTs nanostructure can be deconvoluted to three peaks²¹ centered at 399.39 eV (originated from —NH—), 400.73 eV (originated from =N⁺—), and 402.03 eV (originated from —N⁺—) with area fractions of 48.84%, 23.15%, and 28.12%, respectively. N⁺/N ratio of PANI coatings is 51.15%. The data illuminate that PANI is highly doped by DBSA and is in its emeraldine salt form, MWNTs are wrapped by conducting PANI coatings.

TEM analysis

TEM images of p-MWNTs and PANI-g-MWNTs nanostructure dispersed in ethanol, PANI-g-MWNTs nanostructure, and PANI-g-MWNTs/epoxy resin blending system with 1.0 wt % PANI-g-MWNTs content in THF are shown in Figure 4. As shown in Figure 4(b), p-MWNTs are encapsulated by PANI coatings forming a core-shell nanostructure. When PANI-g-MWNTs nanostructure are dispersed in THF [Fig. 4(c)], PANI chains covalently attached to p-MWNTs are swelling along the surface of p-MWNTs. Because of encapsulated by swelling PANI coatings, MWNTs cannot aggregate forming a stable suspension.²⁰ After epoxy resin is added into the suspension, MWNTs are well dispersed in epoxy

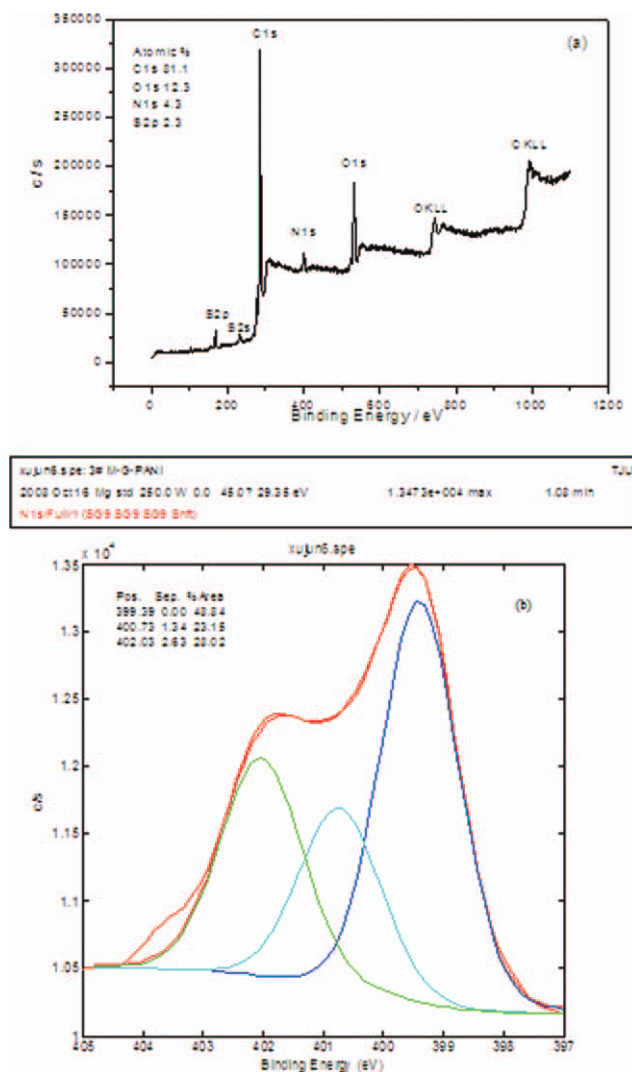


Figure 3 Wide scan XPS spectrum (a) and deconvolution of N1s (b) for PANI-g-MWNTs nanostructure. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

matrix forming a homogenous PANI-g-MWNTs/epoxy resin blending system as can be seen in Figure 4(d).

DSC analysis

DSC curves of neat epoxy and PANI-g-MWNTs/epoxy composite with 1.0 wt % PANI-g-MWNTs content are shown in Figure 5. In the DSC curve of neat epoxy [Fig. 5(a)], curing reaction initiates at 64°C (T_{ia}), finishes at 136°C (T_{fa}), and there is an exothermic peak at 111°C (T_{pa}), which is attributed to epoxy resin curing by EMI-2,4. In the DSC curve of PANI-g-MWNTs/epoxy composite [Fig. 5(b)], curing initial temperature and exothermic peak temperature move to 52°C (T_{ib}) and 108°C (T_{pb}), respectively, and curing reaction finishes at 136°C (T_{fb}). Incorporation of PANI-g-MWNTs nanostructure into epoxy leads to

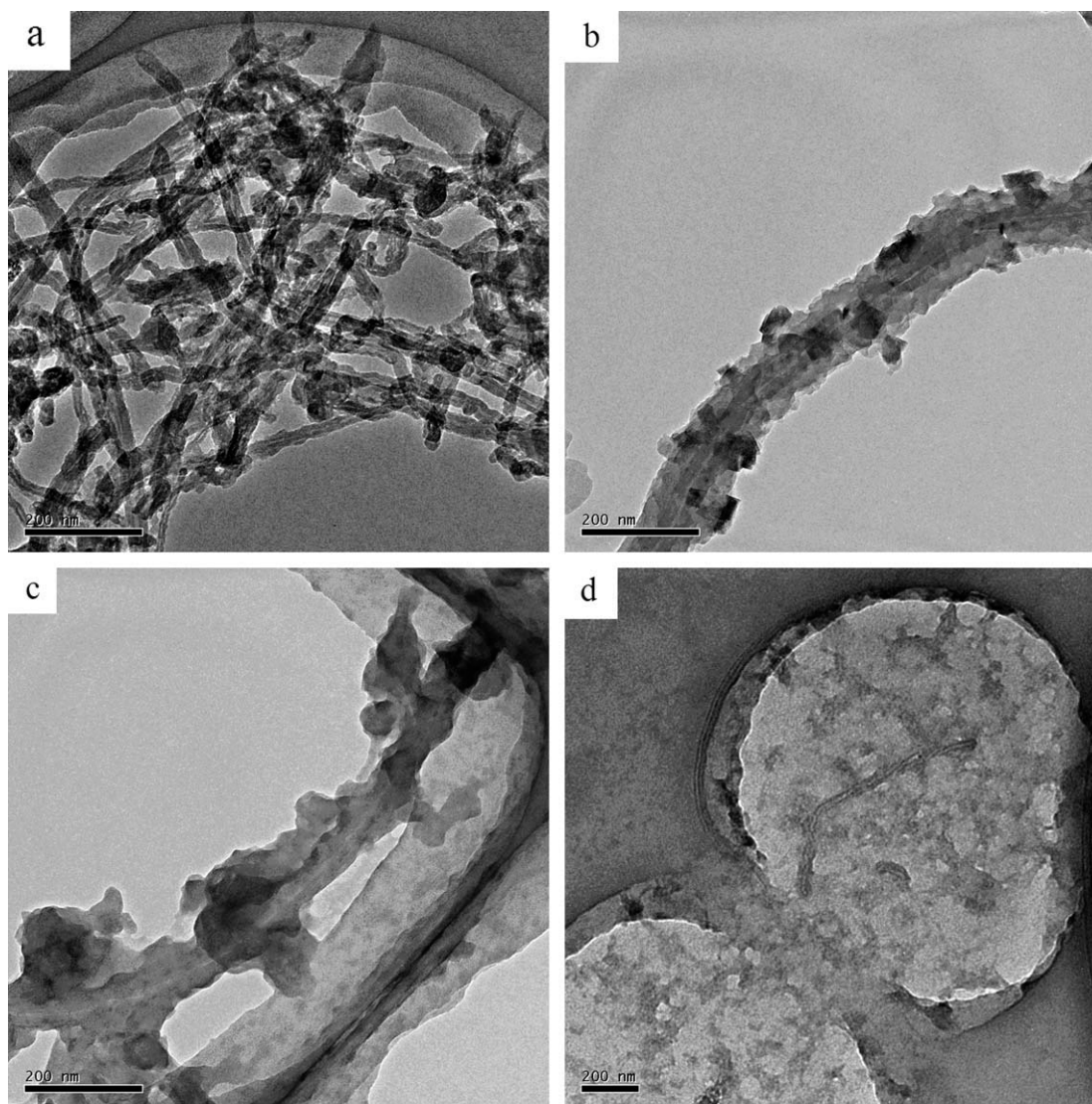


Figure 4 TEM images of p-MWNTs (a) and PANI-g-MWNTs nanostructure (b) in ethanol, PANI-g-MWNTs nanostructure (c), and PANI-g-MWNTs/epoxy resin blending system with 1.0 wt % PANI-g-MWNTs content (d) in THF.

curing reactions occurring at lower temperatures. The results indicate that the PANI-g-MWNTs nanostructure promotes curing reaction of epoxy resin, and terminal amino groups of PANI coatings react with epoxy matrix during curing reactions.

Thermogravimetric analysis

TGA curves of neat epoxy and epoxy composites with different PANI-g-MWNTs contents are shown in Figure 6. Neat epoxy decomposes starting at 297°C and completely decomposes at 450°C. Initial decomposition temperatures of the composites with 0.5 and 1.0 wt % PANI-g-MWNTs contents increase to 306 and 320°C, respectively, which indicates thermal decomposition of the composites are retarded by the presence of PANI-g-MWNTs nanostructures. Value T50 represents the temperature when 50% of

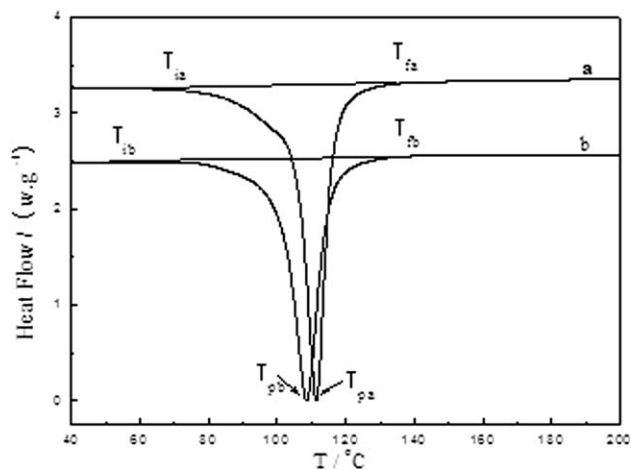


Figure 5 DSC curves of neat epoxy (a) and PANI-g-MWNTs/epoxy composite with 1.0 wt % PANI-g-MWNTs content (b).

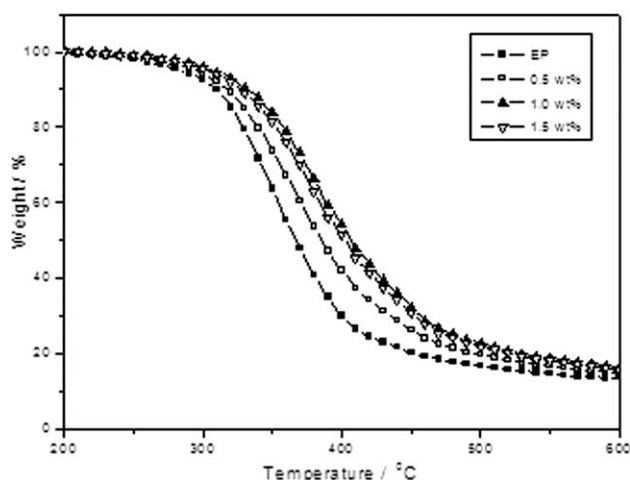


Figure 6 TGA curves of neat epoxy and epoxy composites with different PANI-g-MWNTs nanostructure contents.

original sample is decomposed. Value T50 for neat epoxy is 366°C, and the values increase to 384 and 405°C for the composites with 0.5 and 1.0 wt % PANI-g-MWNTs contents, respectively. These data indicate that thermal stability of the composites is enhanced by incorporation of PANI-g-MWNTs nanostructure into epoxy network. The results may attribute to MWNTs well dispersion in epoxy matrix and MWNTs impede propagation of decomposition reactions in the composites.²² When content of PANI-g-MWNTs is higher than 1.0 wt %, thermal stability of the composite slightly decreases, which may be because of agglomeration of MWNTs in the matrix.⁹

Electrical conductivity

Electrical conductivities at room temperature of neat epoxy and epoxy composites with different PANI-g-MWNTs contents are shown in Figure 7. As shown in Figure 7, electrical conductivity for neat epoxy is $2.848 \times 10^{-13} \text{ S cm}^{-1}$. Incorporation of PANI-g-MWNTs nanostructure increases the conductivity seven orders of magnitude when content of PANI-g-MWNTs is 1.0 wt %, and the conductivity reaches $1.975 \times 10^{-6} \text{ S cm}^{-1}$. The increasing of electrical conductivity implies that percolation threshold for the composites is between 0.25 and 1.0 wt % of PANI-g-MWNTs nanostructure contents. When the loading of PANI-g-MWNTs is higher than 1.0 wt %, the composites behave slow improvement in conductivity. In recent studies, Yuen et al.¹⁵ coated MWNTs with TiO₂ and then modified with 3-(aminopropyl) triethoxysilane (APTES), volume resistivity of the epoxy composite decreased nine orders of magnitude for sample with 0.25 wt % TiO₂-APTES-MWNTs content. Wu et al.²³ treated acid-modified MWNTs with 3-isocyanatopropyltriethoxysilane (IPTES),

volume electrical resistance of the epoxy composite was decreased by six orders of magnitude with 1.0 wt % IPTES-MWNTs content. In our study, we functionalized MWNTs with conjugated DBSA-doped PANI chains, π electron can be transferred through MWNTs walls and PANI coatings, the electrical conductivities at room temperature for p-MWNTs and DBSA-doped PANI-g-MWNTs are 71.69 and $6.23 \times 10^{-1} \text{ S cm}^{-1}$, respectively.²⁰ Because of conducting PANI chains wrapping on the surface of MWNTs, PANI-g-MWNTs possess higher electrical conductivity than the modified MWNTs wrapped by nonconductive molecules. Also, aspect ratio of the core (MWNTs)-shell (PANI) nanofibers is greater than that of regular nanoparticles. Furthermore, because of encapsulated by swelling PANI coatings, MWNTs cannot aggregate and are homogeneously dispersed in epoxy resin, the well dispersion state is in favor of formation of electrical networks.²⁴ As a result, PANI-g-MWNTs/epoxy composites exhibit better electrical conductivity with low quantity of additions.

Mechanical properties

Mechanical properties including tensile strength, Young's modulus, flexural strength, and flexural modulus of the epoxy composites with different PANI-g-MWNTs contents are shown in Figure 8. As shown in Figure 8, both tensile and flexure properties of the composites improve as PANI-g-MWNTs content increases from 0.25 to 1.0 wt %. When content of PANI-g-MWNTs is higher than 1.0 wt %, mechanical properties of the composites behave slowly decreasing. In Figure 8(a), tensile strength is increased by 61 % from 35.26 (neat epoxy) to 56.93 MPa (1.0 wt % PANI-g-MWNTs content), Young's modulus is increased by 43% from 510 (neat epoxy)

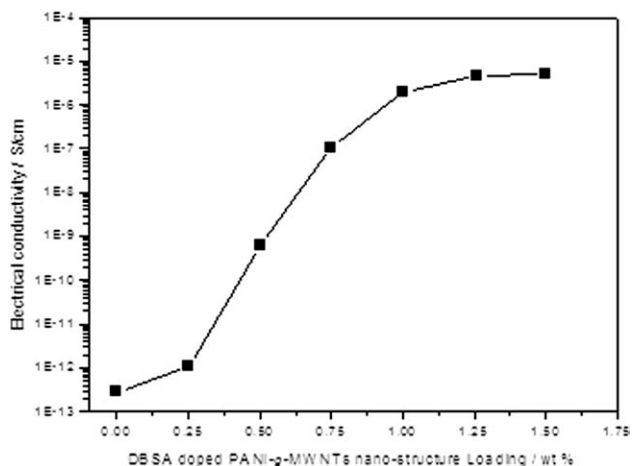


Figure 7 Electrical conductivities of neat epoxy and epoxy composites with different PANI-g-MWNTs nanostructure contents.

to 733 MPa (1.0 wt % content). As the content of PANI-g-MWNTs increases to 1.5 wt %, tensile strength and Young's modulus of the composite decrease to 51.71 Mpa and 681 Mpa, respectively. In Figure 8(b), flexural strength is increased by 78% from 92.99 (neat epoxy) to 165.33 MPa (1.0 wt % content), and flexural modulus is increased by 49% from 1691 (neat epoxy) to 2517 MPa (0.75 wt % content) and to 2510 MPa (1.0 wt % content). As the content of PANI-g-MWNTs increases to 1.5 wt %, flexural strength and flexural modulus of the composite decrease to 153.93 Mpa and 2418 Mpa, respectively. The improved mechanical properties are correlated with strong interfacial adhesion between MWNTs and epoxy resin as well as homogenous dispersion of MWNTs in epoxy matrix. In the PANI-g-MWNTs/epoxy composites, terminal amino groups of PANI coatings react with epoxy matrix during curing reactions, which provide interfacial adhesion for load transfer between epoxy and nanotubes,^{9,25} and therefore, mechanical properties of the composites are enhanced. Also, as shown in Figure 4(d), MWNTs are well dispersed in epoxy resin, which is in favor of exerting performance of

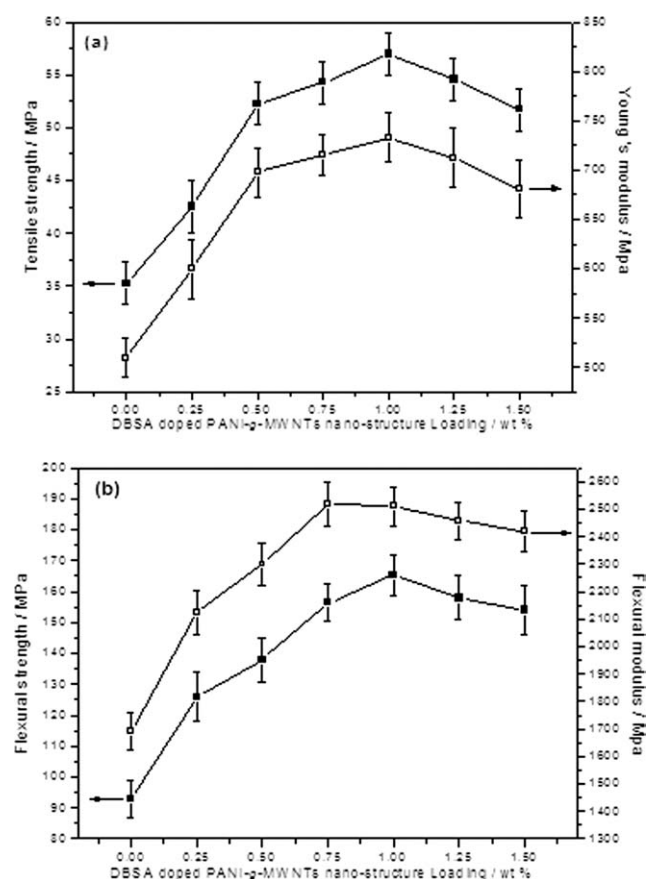


Figure 8 Variations of tensile properties (a) and flexural properties (b) of epoxy composites with PANI-g-MWNTs nanostructure contents.

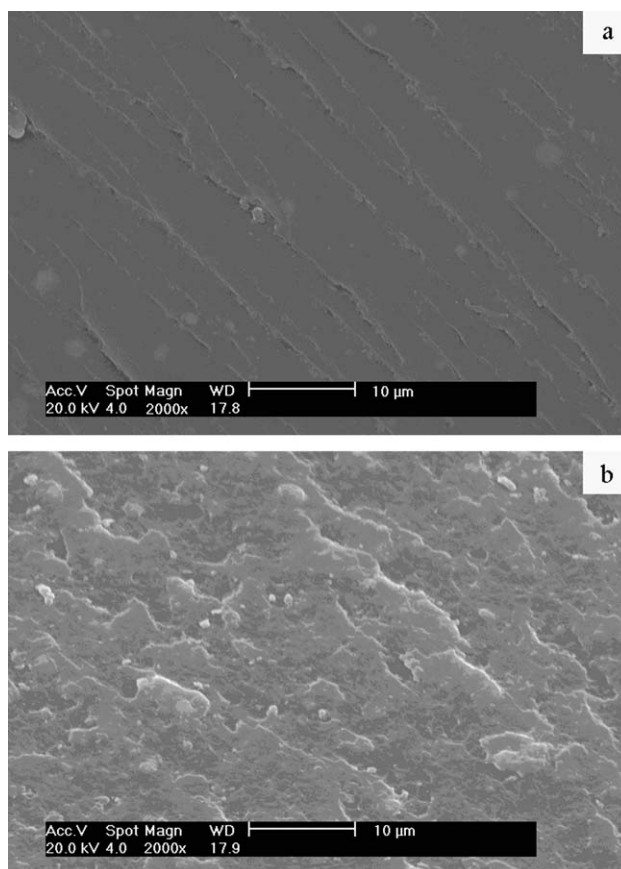


Figure 9 SEM images of fracture surfaces for neat epoxy (a) and PANI-g-MWNTs/epoxy composite with 1.0 wt % PANI-g-MWNTs content (b).

MWNTs in hosting polymer matrix and formation of MWNTs-reinforced epoxy composites.²³

To understand PANI-g-MWNTs influence on mechanical properties of the epoxy composites, morphology of fracture surfaces for neat epoxy and PANI-g-MWNTs/epoxy composite with 1.0 wt % PANI-g-MWNTs content are shown in Figure 9. In Figure 9(a), fracture surface for neat epoxy is smooth and mirror like; there are river marking representing brittle failure of the material. As shown in Figure 9(b), MWNTs are homogeneously embedded in the matrix; they are broken instead of being pulled out. The phenomena indicate existence of strong interfacial bonding between PANI-g-MWNTs and epoxy matrix. Also, fracture surface for the composite become rough and mountain like. It appears that river markings are terminated by well-dispersed MWNTs and that cracks propagate bypass the nanotubes, which results in dissipation more energy when the composite is cracked.⁴

CONCLUSIONS

PANI-g-MWNTs/epoxy composites were prepared by solution blending and mould casting. MWNTs

were encapsulated by conducting DBSA-doped PANI forming a core (MWNTs)-shell (PANI) nanostructure. PANI coatings swelled in THF and MWNTs were homogeneously dispersed in epoxy matrix. With DBSA-doped PANI-g-MWNTs nanostructure introducing into epoxy resin, curing reaction was promoted and thermal stability of the composites was enhanced. Because of conducting PANI chains wrapping on the surface of MWNTs and well dispersion of MWNTs in epoxy matrix, electrical conductivity at room temperature of the composites was increased by seven orders of magnitude compared with neat epoxy. The associated percolation threshold for the composites was between 0.25 and 1.0 wt % of PANI-g-MWNTs loading. In the PANI-g-MWNTs/epoxy composites, terminal amino groups of PANI coatings reacted with epoxy matrix during curing reactions, which guaranteed interfacial adhesion between MWNTs and epoxy resin. Mechanical properties including tensile strength, Young's modulus, flexural strength, and flexural modulus of the composites were increased by 61%, 43%, 78%, and 49% compared with neat epoxy, respectively.

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