

Special Feature: Organic Materials

Research Report

Novel Carbon Nanotube/polymer Composites Having High Thermal Conductivity and Electrical Insulation

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Report received on Dec. 17, 2012

■ABSTRACT■ With their excellent mechanical, electrical, and thermal properties, carbon nanotubes (CNTs) have recently attracted attention as nanofillers that may efficiently improve the physical properties of polymers. However, CNTs cannot practically be used for highly electrically insulating polymers because even a few CNTs impart a high electrical conductivity to the polymers. In the present study, we design and propose a novel morphological model for CNT/polymer composites with high thermal conductivity and electrical insulation. In the proposed morphology, the CNT/polymer composites are composed of a matrix polymer and a CNT-localizing domain polymer encapsulated by a shell-forming component, which contributes to the selective localization of the CNTs into the dispersed domains. Furthermore, based on the proposed model, in which the CNT-localizing regions are intrinsically controlled by the higher affinity of the domain polymer to the CNTs compared to that of the matrix polymer, we have fabricated CNT/poly(phenylene sulfide)-based composites having high thermal conductivity and electrical insulation. In engineering applications of the proposed model, the CNT/polymer composites used as the polymer matrix are expected to efficiently provide outstanding thermal conductivity and high electrical insulation at lower loadings of thermally conductive inorganic fillers.

■KEYWORDS■ Carbon Nanotube, Polymer Blend, Composite, Morphology, Thermal Conductivity, Electrical Insulation, Inorganic Filler

1. Introduction

With recent widespread of hybrid electric vehicles (HEVs) and electric vehicles (EVs), as well as forthcoming fuel cell vehicles (FCVs), the need to efficiently diffuse heat from inside electric and electronic devices has become increasingly important because of trends in significant size reduction and high-power design from the viewpoint of CO₂ emission reduction. Owing to their remarkable electrical insulation characteristics, low density (lightweight), and high melt-molding processability, polymers are predominantly used as insulating materials in the above-mentioned devices. As shown in **Fig. 1** for example, insulating polymer components or layers are generally arranged between electrical components that cause high heat generation, and metal heat sink components, and efficient heat transfer from the polymer components to the heat sink components is strongly expected. However, the thermal conductivity of polymers is considerably low ($\sim 0.2 \text{ W m}^{-1} \text{ K}^{-1}$). Therefore, improving their thermal conductivity is imperative. In order to enhance their

thermal conductivity, composite materials consisting of polymers and thermally conductive inorganic fillers, such as alumina (Al₂O₃) and boron nitride (BN), are generally used. The incorporation of inorganic fillers into the polymer matrix, however, results in weight increase, lower viscoelasticity, lower melt-molding processability (high viscosity), and increased costs.

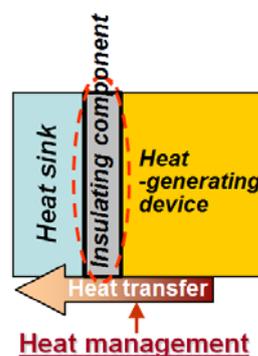


Fig. 1 Schematic diagram of insulating polymer arranged between heat-generating device and metal heat sink.

Owing to their extremely high thermal conductivity ($\sim 3,000 \text{ W m}^{-1} \text{ K}^{-1}$),⁽¹⁻³⁾ carbon nanotubes (CNTs), have recently attracted attention as effective nanofillers, which will enhance the thermal conductivity of polymers.⁽⁴⁻¹¹⁾ Therefore, the incorporation of CNTs in low concentrations ($\sim 1 \text{ vol}\%$) into polymers has been considered to be an effective method of improving the thermal conductivity of polymers without adversely affecting their advantageous properties, as estimated through an effective medium approach combined with Kapitza's thermal contact resistance concept.⁽¹²⁾ However, CNTs cannot practically be used for highly electrically insulating polymers because even a few CNTs impart a high electrical conductivity to the polymers.^(4-9,13,14) We herein describe CNT/polymer composites having a novel morphology that provides both enhanced thermal conductivity and high electrical insulation.

2. Morphological Model for CNT/polymer Composites with High Thermal Conductivity and Electrical Insulation

CNT/polymer composites exhibit electrical conductivity at low CNT concentrations (less than several vol%) above the percolation threshold, forming CNT conductive paths in polymer matrices.^(4-9,13,14) We propose a morphological model for CNT/polymer composites having high thermal conductivity and electrical insulation, as shown schematically in **Fig. 2**. The proposed model consists of (i) CNTs; (ii) a matrix polymer; (iii) a domain polymer, which is not compatible with the matrix polymer and has a higher affinity for CNTs compared to the matrix polymer; and (iv) a shell-forming component, which acts as both a

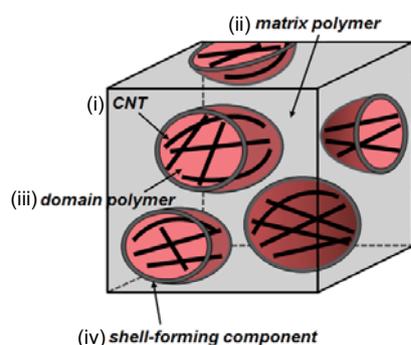


Fig. 2 Schematic diagram of novel morphology of CNT/polymer composites having high thermal conductivity and electrical insulation.

stabilizing agent at their interface and as a repulsive agent for the CNTs. The CNTs are selectively localized within the domain polymers by both their high affinity for the domain polymers and the existence of the shell-forming component (i.e., shell-layer) at the matrix polymer-domain polymer interfacial region. The shell-forming component is required to have high affinity for both the matrix and domain polymers and low affinity (repulsive effects) for the CNTs in order to lock up the CNTs into the domain polymer, preventing the formation of electrical conductive paths of the CNTs in the polymer matrix. In the present study, CNT/PPS-based (matrix) composites were prepared for the purpose of experimentally investigating the validity of the proposed morphology, as shown in **Fig. 2**.

3. Preparation and Properties of CNT/PPS-based Composites

3.1 Materials

Multi-walled CNTs (MWCNT) (Nano Carbon Technologies Co., Ltd., Japan, MWNT-7, diameter: 40 to 90 nm, purity > 99.5 wt%) were used without any further purification. Poly(1,4-phenylene sulfide) (PPS) (Sigma-Aldrich Co., melt viscosity (310°C , $1,200 \text{ s}^{-1}$): 27.5 Pa s , specific gravity: 1.35), high-density polyethylene (PE) (Japan Polyethylene Co., Novatec HD HY430, specific gravity: 0.96) and poly(ethylene-co-glycidyl methacrylate) (EGMA) (Sumitomo Chemical Co., Ltd., Bondfast E: glycidyl methacrylate content: 12 wt%, specific gravity: 0.94) were used as polymer components. PPS, which exhibits both high electrical insulation and high heat resistance suitable for electric and electronic devices, was used as the main polymer component forming the continuous phase (matrix), and PE was selected as the polymer component forming the dispersed phase (domain). Spherical BN filler (FS-3, Mizushima ferroalloy Co., Ltd., Japan, particle size: $\sim 50 \mu\text{m}$, specific gravity: 2.26) was used as a typical thermally conductive inorganic filler.

3.2 Preparation of CNT/polymer Composites

Dry blends of MWCNTs (1 vol%), PPS (71 vol%), PE (21 vol%), and EGMA (7 vol%) were fed into a twin-screw extruder (Technovel Co., KZW15-60MG, screw diameter (D): 15 mm, screw length/diameter (L/D): 60), melt-compounded at a processing

temperature of 290°C with a constant rotation rate of 100 rpm, extruded into strands and subsequently pelletized after water cooling. Pellets of the MWCNT/PPS-based composites were obtained for test specimens after drying under a vacuum at 130°C for 6 h. Test specimens (25 mm × 25 mm, thickness: 1 mm or 2 mm) of the obtained composites were prepared by compression molding at 300°C.

3.3 Morphology of CNT/PPS-based Composites

For observations of morphologies of the obtained MWCNT/PPS-based composites, a transmission electron microscope (TEM) image of an ultrathin section was obtained using a JEOL JEM-2000EX instrument operated at 200 kV. Scanning electron microscope (SEM) images of frozen fracture surfaces of the test specimens were obtained using a Hitachi S-3600N SEM.

Figure 3 shows a TEM image of the MWCNT/PPS/

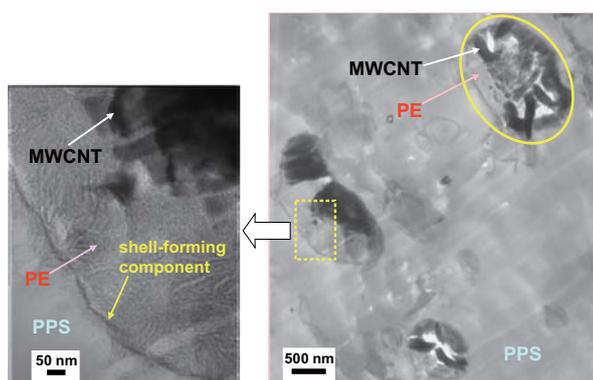


Fig. 3 TEM images of MWCNT/PPS/PE/EGMA (1 vol% / 71 vol% / 21 vol% / 7 vol%) composite.

PE/EGMA composites. This image shows that the MWCNTs were selectively localized in dispersed PE domains. The size of the PE domains was on the micrometer scale. The amorphous regions in PE were observed in high-contrast TEM images using ruthenium tetroxide⁽¹⁵⁾ as a selectively staining agent. As shown in the higher-magnification TEM image in Fig. 3, the shell-forming component located between the dispersed PE domain and the continuous PPS matrix was observed as a mostly stained region. This highly stained region is believed to be assigned to an amorphous EGMA component existing in the interfacial region between PE and PPS.⁽¹⁶⁾

In the present study, PE was selected as the polymer component forming the dispersed phase (domain) because of the higher affinity between PE and the MWCNTs, as compared to that between PPS and the MWCNTs, associated with an SEM image showing the morphology of MWCNT (1 vol%)/PPS (71 vol%)/PE (28 vol%) composites (**Fig. 4(a)**). Previous studies have shown that, in general, CNTs could be highly dispersed in the PE phase through polymer-CNT interactions⁽¹⁷⁾ by melt-blending.^(18,19) Thus, the morphology in which most of the MWCNTs are localized in the PE domains is considered to be formed through these interactions and does not exhibit initial aggregates of MWCNTs in the PE phase. The shell-layer in this morphology is considered to be formed from EGMA, in which the ethylene units have good affinity for PE and epoxy groups in glycidyl methacrylate units can react with the terminal groups of PPS as a compatibilizer.⁽¹⁶⁾ For the MWCNT (1 vol%)/PPS (matrix: 71 vol%)/EGMA (dispersed domains: 28 vol%) composites shown in Fig. 4(b), MWCNTs were not included in the EGMA domains,

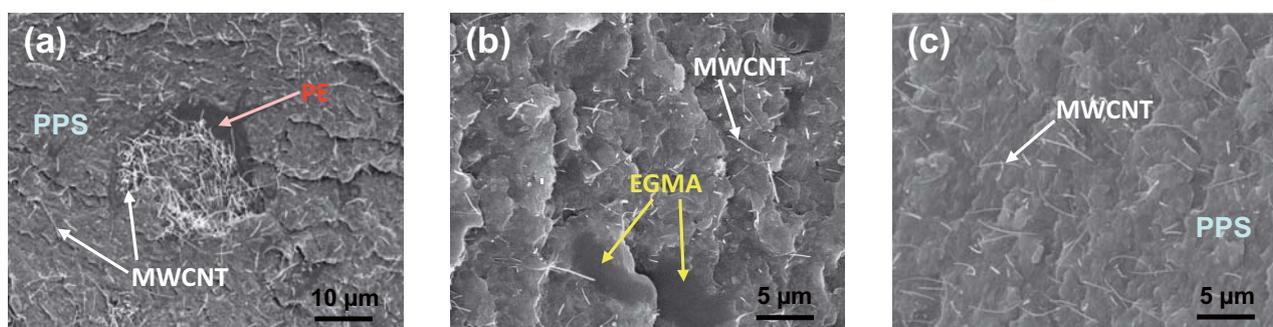


Fig. 4 SEM images of CNT/PPS-based composites: (a) MWCNT/PPS/PE (1 vol% / 71 vol% / 28 vol%), (b) MWCNT/PPS/EGMA (1 vol% / 71 vol% / 28 vol%), and (c) MWCNT/PPS (1 vol% / 99 vol%).

which have a relatively low affinity for the MWCNTs. For the MWCNT (1 vol%)/PPS (99 vol%) composites shown in Fig. 4(c), the MWCNTs were uniformly dispersed in the PPS matrix by simple melt-blending, inferring that numerous instances of physical contact occur between CNTs in the PPS matrix, reducing electrical insulation.

3.4 Physical Properties of CNT/PPS-based Composites

Electrical Insulation

Volume resistivity was measured for the compression molded test specimens (disc-like samples having diameters of 100 mm and thicknesses of 1 mm) using a high-resistance meter (Agilent Technologies, AGILENT 4339B). A potential of either 1,000 V or 100 V was applied to the specimen, and the volume resistivity was measured after 20 s. The average of four measurements was regarded as volume resistivity. The breakdown electric field of electrically conductive paste (Fujikura Kasei Co., Ltd., Dotite FA-705BN)-attached compression molded test specimens was measured using a withstanding voltage tester (Kikusui Electronics Co, TOS 8750, DC mode).

The volume resistivity of the obtained MWCNT/PPS/PE/EGMA (1 vol%/71 vol%/21 vol%/7 vol%) composites after the application of a potential of 1,000 V was evaluated to be $3.3 \times 10^{15} \Omega \text{ cm}$, which is comparable to that of pristine PPS ($3.4 \times 10^{15} \Omega \text{ cm}$), and exhibited an electrical insulation that was in accordance with the morphology shown in Fig. 3. The MWCNT/PPS (1 vol%/99 vol%), MWCNT/PPS/PE (1 vol%/71 vol%/28 vol%), and MWCNT/PPS/EGMA (1 vol%/71 vol%/28 vol%) composites exhibited an overcurrent at 1,000 V, with volume resistivities at 100 V of $2.4 \times 10^{13} \Omega \text{ cm}$, $2.9 \times 10^9 \Omega \text{ cm}$, and $3.9 \times 10^{13} \Omega \text{ cm}$, respectively (Fig. 5). In addition, for MWCNT/PPS/PE/EGMA composites, the breakdown electric field of was evaluated using a withstand voltage tester. Test specimens of the MWCNT/PPS/PE/EGMA composites of 1 mm thickness did not show any dielectric breakdown even at 5.0 kV, which is the maximum voltage that can be applied by this tester. Therefore, the breakdown electric field of MWCNT/PPS/PE/EGMA composites was found to be $>5.0 \text{ kV mm}^{-1}$. In contrast, the breakdown electric field of MWCNT/PPS composites was 0.6 kV mm^{-1} .

Moreover, the insulating properties of the MWCNT/PPS/PE/EGMA and MWCNT/PPS composites

were investigated on a nanometer scale by detecting minute currents using a scanning probe microscope (SPM). Detection of minute currents ($< \text{pA}$ level) was carried out using Veeco Dimension V equipped with a TUNA module. Figure 6 shows a schematic diagram of the minute current measurements and minute current images for both of the composites. The cross-section of the compression molded test specimen was polished to create a relatively smooth surface. The sample was coated with gold on one side and was scanned using a Pt-coated probe (Fig. 6(a)). No minute current was observed for the MWCNT/PPS/PE/EGMA composites with a direct current (DC) bias of 10 V. For the MWCNT/PPS composites, electrically conductive paths were observed even with a DC bias of 500 mV (Fig. 6(b)).

For the MWCNT/PPS composites in which the MWCNTs were uniformly dispersed in the PPS matrix, as shown in Fig. 4(c), physical contacts between the MWCNTs might occur above the percolation threshold, followed by the loss of electrical insulation. In contrast with the MWCNT/PPS composites, for the MWCNT/PPS/PE composites, most of the MWCNTs were located in the PE domains, but still appeared in the PPS matrix in appreciable quantities, as shown in Fig. 4(a). This morphology is considered to follow the formation of electrically conductive paths, leading to a drastic decrease in volume resistivity (Fig. 5). Based on these results, as a shell-forming component exhibiting low affinity for

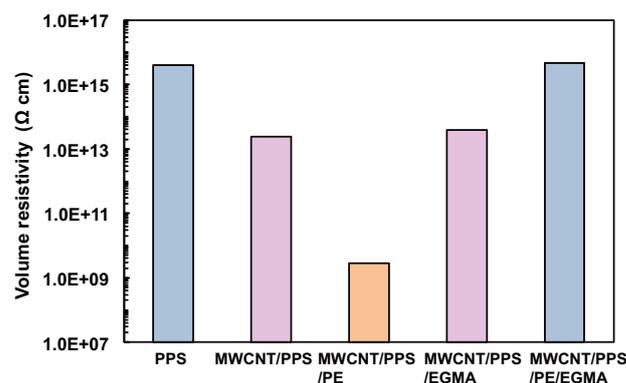


Fig. 5 Volume resistivity of PPS, MWCNT/PPS (1 vol% / 99 vol%) composites, MWCNT/PPS/PE (1 vol% / 71 vol% / 28 vol%) composites, MWCNT/PPS/EGMA (1 vol% / 71 vol% / 28 vol%) composites, and MWCNT/PPS/PE/EGMA (1 vol% / 71 vol% / 21 vol% / 7 vol%) composites after applying a potential of 100 V.

MWCNTs, EGMA contributes to selective localization of the MWCNTs in the PE domains without forming electrically conductive paths in the PPS matrix.

Thermal Conductivity

The cross-section of the compression molded test specimen (25 mm × 25 mm, thickness: 2 mm) was polished to create a relatively smooth surface. The thermal conductivity of each specimen was measured at 40°C using a steady-state technique. The thermal conductivity of the MWCNT/PPS/PE/EGMA (1 vol%/ 71 vol%/21 vol%/7 vol%) composites increased drastically compared with pristine PPS or PPS/PE/EGMA (72 vol%/21 vol%/7 vol%) composites, even at a very low MWCNT loading (1 vol%), as shown in Fig. 7. The thermal conductivity of the MWCNT/PPS/PE/EGMA composites ($0.43 \text{ W m}^{-1} \text{ K}^{-1}$) was approximately 1.8 times higher than that of pristine PPS ($0.24 \text{ W m}^{-1} \text{ K}^{-1}$). In addition, the thermal conductivity of the MWCNT/PPS/PE/EGMA composites was much higher than that of the MWCNT/PPS composites ($0.36 \text{ W m}^{-1} \text{ K}^{-1}$) in which

the MWCNTs were uniformly dispersed in the PPS matrix, although theoretical estimates of their thermal conductivities based on conventional approaches^(12,20,21) were low (approximately $0.3 \text{ W m}^{-1} \text{ K}^{-1}$). Previous attempts to improve the thermal conductivity of polymers have focused on forming percolated paths of CNTs in a polymer matrix,⁽⁴⁻⁹⁾ which work well for electrical conductivity. However, the proposed morphology for the CNT/polymer composites was confirmed to be very effective for enhancing the thermal conductivity of CNT/polymer composites without forming the percolated paths in the polymer matrix. In general, the major obstacle in using CNTs to improve thermal conductivity of the CNT/polymer composites is phonon scattering at the CNT-polymer interface.^(6,7,9,11) Thus, it is reasonable to consider that, for the prepared MWCNT/PPS/PE/EGMA composites, interfacial phonon scattering between MWCNT-polymer interfaces is likely to be reduced by a decrease in the MWCNT-polymer interfacial area, due to the occurrence of local contacts of MWCNTs limited in the PE-dispersed domains. In addition, the interfacial phonon scattering between the PE-dispersed domain and the PPS matrix is also considered to be reduced by the existence of shell layers formed through the reaction of EGMA in the shell-forming component with the terminal groups of PPS.

3.5 CNT/PPS-based Composites with Thermally Conductive Inorganic Fillers

Furthermore, the proposed morphology can be also

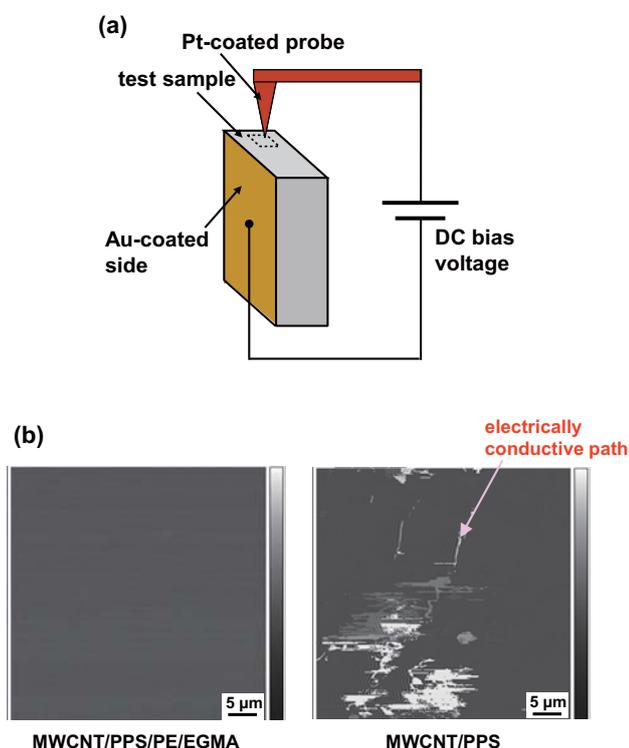


Fig. 6 (a) Schematic diagram of detection of minute current by SPM. (b) Minute current images of MWCNT/PPS/PE/EGMA composite (DC bias: 10 V) and the MWCNT/PPS composite (DC bias: 500 mV) detected by SPM.

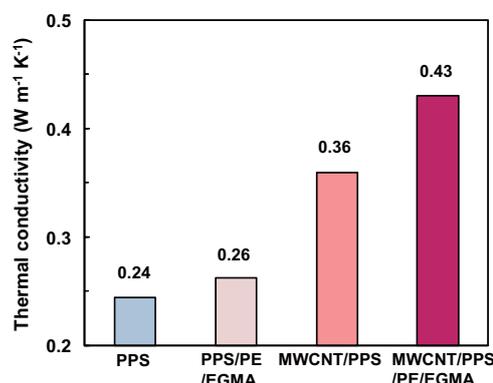


Fig. 7 Thermal conductivity of PPS, PPS/PE/EGMA (72 vol% / 21 vol% / 7 vol%) composites, MWCNT / PPS (1 vol% / 99 vol%) composites, and MWCNT/PPS/PE/EGMA (1 vol% / 71 vol% / 21 vol% / 7 vol%) composites.

applied to efficiently enhance the thermal conductivity of polymers filled with thermally conductive inorganic fillers. As thermally conductive inorganic fillers, the effects of BN particle loading on the thermal conductivity of the PPS-based composites were significantly amplified by approximately the thermal conductivity ratio of CNT-filled matrix/unfilled matrix in the presence of the MWCNTs without affecting electrical insulation (Figs. 8 and 9). These results show that the increase in the thermal conductivity of polymers associated with the introduction of the proposed morphology imparts greater thermal conductivity to polymer/inorganic filler composites at a low filler loading.

Figure 10 shows the effects of the CNT/PPS-based

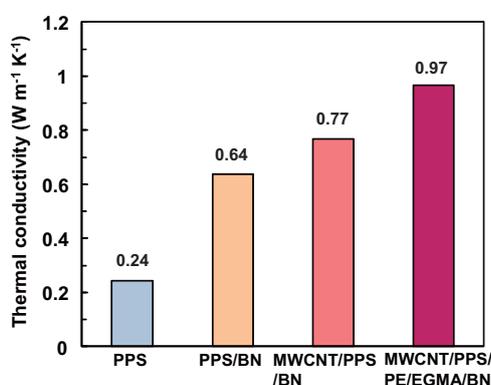


Fig. 8 Thermal conductivity of PPS, PPS/BN (85 vol% / 15 vol%) composites, MWCNT/PPS/BN (0.85 vol% / 84.15 vol% / 15 vol%) composites, and MWCNT/PPS/PE/EGMA/BN (0.85 vol% / 60.35 vol% / 17.85 vol% / 5.95 vol% / 15 vol%) composites.

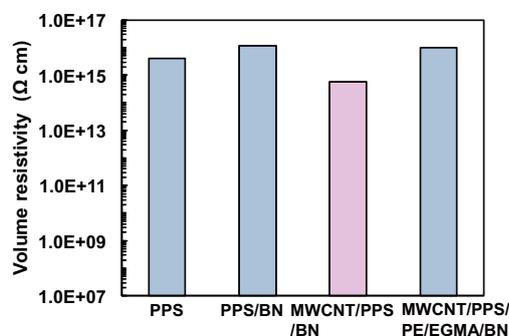


Fig. 9 Volume resistivity of PPS, PPS/BN composites, MWCNT/PPS/BN composites, and MWCNT/PPS/PE/EGMA/BN composites after applying potential of 100 V.

composite matrix on insulating polymer systems with thermally conductive inorganic fillers for two engineering applications (A and B). For application A, namely, the use of the CNT/PPS-based composite matrix at the same loading of the inorganic fillers, it is expected that enhanced thermal conductivity of the insulating polymer system can be easily attained through thermal conductivity amplification effects of the composite matrix. For application B, namely, the use of the CNT/PPS-based composite matrix at a lower loading of inorganic fillers, the same level of the thermal conductivity of the insulating polymer system can be attained with lower densities, improved melt-molding processability, and possibly lower material costs.

4. Application of the Proposed Model to CNT/other Polymer-based Composites

For MWCNT/PPS matrix/PE domain composites, in which the PE component has higher affinity for MWCNT, as compared to the PPS component, the CNTs are selectively localized within the PE domains dispersed in the PPS matrix. Furthermore, in the present study, the validity of the proposed morphological model, which exhibits enhanced thermal conductivity without loss of high electrical insulation, as shown in Fig. 2, has been generally confirmed based on experimental results for other polymer matrix systems, i.e., MWCNT/polyamide-6 (PA6)-based composites. Multi-walled CNTs (1 vol%), PA6 (71 vol%), PPS (21 vol%), and a shell-forming polymer (7 vol%) were blended for the preparation of MWCNT/PA6-based composites using a twin-screw

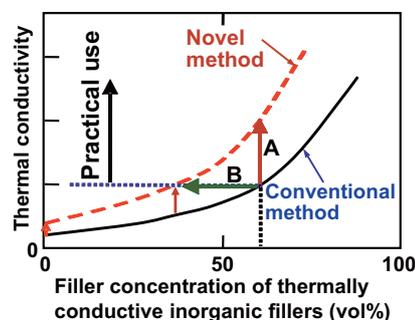


Fig. 10 Amplification effects of λ_m for CNT/polymer composites with thermally conductive fillers. Arrows A and B indicate different purposes for use of CNT/polymer composite matrix.

extruder in a manner similar to that used to obtain the MWCNT/PPS-based composites.

Figure 11 shows an SEM image of the MWCNT/PA6/PPS/shell-forming polymer composites. The CNTs are selectively localized within the PPS domains dispersed in the PA6 matrix under the condition that the domain polymer (PPS) has a higher affinity for the CNTs, as compared to the matrix polymer (PA6). **Figure 12** shows the thermal conductivity and volume resistivity for pristine PA6 and MWCNT/PA6/PPS/shell-forming polymer composites. The figure shows that the MWCNT/PA6-based composites exhibits high thermal conductivity and high electric insulation in a manner similar to the MWCNT/PPS-based composites. Thus, for MWCNT/polymer-based composites with high thermal conductivity and high electrical insulation, it has been experimentally confirmed that the MWCNT-localizing regions in the controlled morphology shown schematically in Fig. 2 are intrinsically determined by the relative difference in the affinity for MWCNTs between different polymer components.

5. Conclusions

We have designed and fabricated CNT/polymer composites having a novel morphology consisting of a PPS matrix and CNT-localized PE domains encapsulated by shell-layers formed from a shell-forming component. We demonstrated that such a controlled morphology could achieve both enhanced thermal conductivity and high electrical insulation. To our knowledge, this is the first effective demonstration of using CNTs to achieve enhanced thermal

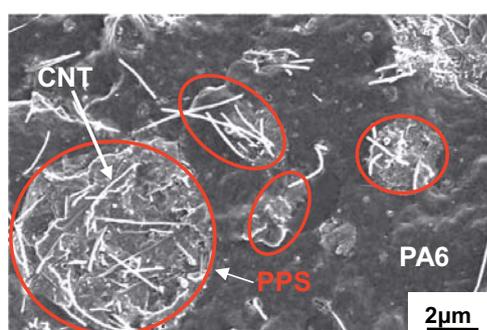


Fig. 11 SEM image of obtained MWCNT/PA6/PPS/shell-forming component (1 vol% / 71 vol% / 21 vol% / 7 vol%) composite.

conductivity of polymer composites with high electrical insulation. Similar morphologies could be easily fabricated using various combinations of polymers by appropriately controlling the affinity between CNTs and each constituent polymer. The proposed morphology represents a promising means by which to realize various applications of CNTs in materials requiring high electrical insulation. For electrically insulating polymers in various electrical and electronic devices, the CNT/polymer composites used as the insulating polymer matrix are expected to efficiently provide outstanding thermal conductivity and high electrical insulation at lower loadings of thermally conductive inorganic fillers while providing characteristics such as low density and high melt-molding processability.

Acknowledgements

The authors would like to thank Ohta, T., Takahara, M., Suzuki, N., Mitsuoka, T. and Koga, T. of Toyota Central R&D Labs., Inc. for their helpful discussions and characterization of the composites.

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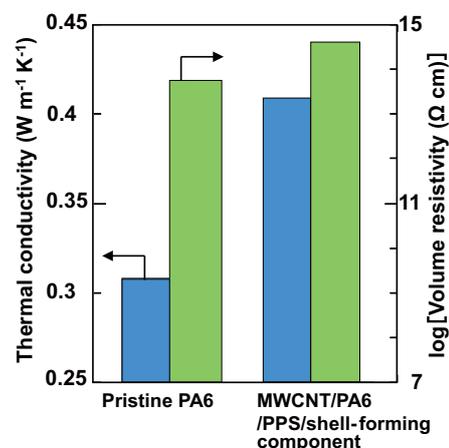


Fig. 12 Thermal conductivity and volume resistivity for PA6 and MWCNT/PA6/PPS/shell-forming component (1 vol% / 71 vol% / 21 vol% / 7 vol%) composites.

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Figs. 2-9

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