Research Article

Linear Assemblies of BN Nanosheets, Fabricated in Polymer/BN Nanosheet Composite Film

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Linear assemblies of BN nanosheets (LABNs) were fabricated in polysiloxane/BN nanosheet composite film under a high DC electric field. The hexagonal BN nanosheets were dispersed by sonication in a prepolymer mixture of polysiloxane followed by a high-speed mixing. The homogeneous suspension was cast on a spacer of microscale thickness and applied to a high DC electric field before it became cross-linked. X-ray diffraction, scanning electron microscopy, and digital microscopy revealed that LABNs formed in the polysiloxane matrix and that the BN nanosheets in the LABNs were aligned perpendicular to the film plane with high anisotropy. This is the first time that linear assemblies of nanosheets have been fabricated in an organic-inorganic hybrid film by applying a DC electric field. The enhanced thermal conductivity of the composite film is attributed to the LABNs. The LABN formation and heat conduction mechanisms are discussed. The polysiloxane/BN nanosheet composite film has the potential to be used semiconductor applications that require both a high thermal conductivity and a high electric insulation.

1. Introduction

Methods for aligning nanosheets in polymer/nanosheet composites have received considerable interest because the physical properties of such composites can be enhanced when the nanosheets are suitably aligned in the polymer matrix [1–6]. In particular, the deformation, failure, heat resistance, and thermal properties of the polymer can be controlled by adding small quantities of hard inorganic particles. Well-aligned nanosheets with a one-dimensional orientation in a polymer matrix exhibit conductivity percolation at a much lower volume fraction than other powders [7–9]. Composites of anisotropically aligned graphite nanosheets in polymers have been attracting considerable interest since such composites exhibit high thermal and electrical conductivities [1, 6–8]. However, these composites have limited application as electrical insulators because graphite exhibits electrical properties that range from metallic to semiconducting [2–4]. Boron nitride (BN) is a promising

material for replacing graphite for applications that require insulators. It has one of the highest thermal conductivities of all electric insulators [10] and exhibits exceptional semiconducting properties with high band gap energies that vary in range 5.5 to 6.4 eV depending on the polymorph and superior chemical inertness. The thermal conductivity of hexagonal BN increases with increasing anisotropy: when BN nanosheets are aligned perpendicular to the *c*-axis, their thermal conductivity is almost 20 times greater than that when they are aligned parallel to the *c*-axis [10, 11]. Aligning nanosheets by reorientation in a polymer is an important technique; shear forces [12], magnetic forces [1, 2, 13], and electric fields [3, 4, 14] have been widely used to reorient nanosheets in a polymer matrix. Shear-induced assembly can align nanosheets in a polymer matrix without surface modification, but it cannot be used to orient nanosheets perpendicular to the composite film surface [12]. Orientating nanosheets using electric fields and magnetic forces normally requires modifying the nanosheet surface

by adding metal nanoparticles (e.g., iron nanoparticles) to achieve better orientation of the nanosheets [2, 3]. High torques, generated using nanosecond electrical pulses with potentials up to 40 kV, have been applied to avoid surface modification [15]. Orientating objects such as nanoparticles [16], nanotubes [17], and nanosheets [14, 17–19] in a polymer matrix by applying an electric field has been investigated to determine the orientation mechanisms in terms of the structure variation. Linear bundles of carbon nanotubes have been fabricated in organic solvents by applying a DC electric field [17]. Such elongated bundle structures are interesting because, unlike one-dimensional nanotube structures, two-dimensional structures enable nanosheets to be used as conducting fillers. However, no studies have investigated fabricating linear bundles of nanosheets in a solvent or a viscous polymer. Furthermore, there have been very few studies on fabricating ordered BN nanosheet-polymer composite films [15, 20] and there have been no detailed investigations on the dependence of the physical properties of composites on the arrangement of BN nanosheets.

The three objectives of the present study were to fabricate linear assemblies of BN nanosheets (LABNs) in a polymer/nanosheet composite film by applying a high electric field without modifying the surface, to determine the physical properties of the composite, and to clarify the dependence of the physical properties on the arrangement of the BN nanosheets in the polymer matrix. BN nanosheets homogeneously dispersed in a prepolymer mixture of polysiloxane were subjected to a high DC electric field during cross-linking of the system. X-ray diffraction (XRD), scanning electron microscopy (SEM), digital microscopy, and thermal conductivity measurements were used to characterize the composites.

2. Experimental

2.1. Materials. Polysiloxane/BN nanosheet composite films were prepared by introducing BN nanosheets into a polysiloxane prepolymer mixture. Hexagonal BN nanosheets ($D_{90} = 10.6 \mu\text{m}$, density: 2.26 g/cm^3 , thickness: $2\text{--}10 \text{ nm}$) of commercial origin (Denka Co., Ltd.) were used. Two polysiloxane prepolymers with different viscosities were used: YE5822(A) (viscosity: $1.2 \text{ Pa}\cdot\text{s}$), and YE5822(B) (viscosity: $0.2 \text{ Pa}\cdot\text{s}$) (Momentive Performance Materials Inc.).

2.2. Fabrication of Ordered Polysiloxane/BN Composite Films by Applying a High DC Electric Field. Polysiloxane/BN nanosheet composites were prepared by the following method. 3 g of silicone YE5820(A) was sonicated for 5 min. 0.3 g of silicone YE5822(B) and 0.416 g of BN (5 vol%) were mixed, introduced into the sonicated silicone YE5820(A) and further sonicated for 10 min. The mixture was stirred using a high-speed mixer at 1500 rpm for 5 min to produce a homogeneous dispersion. It was then cast on a glass spacer ($1.2 \text{ mm} \times 1.2 \text{ mm} \times 120 \mu\text{m}$) and subjected to a DC electric field (2.5 kV) for 16 h to enhance the orientation of the BN nanosheets in the polysiloxane prepolymer mixtures perpendicular to the electrodes. Finally, the prepared composites were dried for 0.5 h at 80°C to ensure complete curing.

2.3. Characterization and Measurements. The anisotropic alignment of BN nanosheets in the polymer films were analyzed by XRD (RINT 2500, Rigaku Co.). Reflections from the BN nanosheets were observed at $2\theta = 26.76^\circ$ for the (002) plane and at $2\theta = 41.60^\circ$ for the (100) plane. The linear distribution of BN nanosheets in a polysiloxane matrix was observed by digital microscope (VHX-9000, Keyence Co.). Cross-sections of the polymer/BN composite films were then cut and the surface morphologies of the composites were observed by SEM (JSM-6700F, JEOL Ltd.). The thermal conductivities of prepared composites were analyzed using a thermal diffusivity measurement system that is based on temperature wave analysis (ai-Phase Co., ai-Phase Mobile 1).

3. Results and Discussion

3.1. Orientation and Linear Assembly of BN Nanosheets. The relocation of nanoparticles in a suspension by an external torque force is very sensitive to particle size and bulkiness [21, 22]. In particular, when controlling the anisotropy of nanosheets in a viscous polymer by electrophoresis, nanosheets experience a high reversing force due to forces such as viscoelastic forces and a higher shear force than that exerted on nanorods or nanotubes [20]. Therefore, it is important to select BN nanosheets that are small and have high aspect ratios. Figure 1 shows SEM images of hexagonal BN nanosheets with diameters in the range $10\text{--}20 \mu\text{m}$ and thicknesses in the range $2\text{--}10 \text{ nm}$; these nanosheets exhibited the highest electrophoretic response in water of all the nanosheets fabricated in a previous study by us [15]. The SEM images reveal that the BN nanosheets have graphite-like planar structures and that some of them have agglomerated, whereas others exist as single sheets. The BN nanosheets have smooth surfaces and curved edges.

As a modified graphite nanosheets and the prepolymer mixture are subject to a magnetic field, the graphite nanosheets orientate themselves to minimize the magnetostatic energy and to overcome the free energy (i.e., Brownian motion) of the system until they form a stable configuration [1]. Nanosheets become polarized in an electrical field, which results in a field-induced torque T acting on the sheet, which is given by

$$T = \mu \times E, \quad (1)$$

where μ is the polarization moment and E is the electric field strength. The polarization can be divided into two contributing components: one parallel to the flake ($\mu_{\parallel}$) and one perpendicular to the flake ($\mu_{\perp}$). The torque orients the graphite parallel to the electric field and it opposes the viscous drag of the resin matrix [20, 23]. The total torque acting on the graphite flake can be expressed as a superposition of the torques due to the electric field which are parallel and perpendicular to its flake, $T = \mu_{\parallel} \times E_{\perp} + \mu_{\perp} \times E_{\parallel}$ (where $E_{\parallel} = E \cdot \cos \theta$ and $E_{\perp} = E \cdot \sin \theta$). This can be written as

$$T = \frac{V}{2} \epsilon_0 \epsilon_2 \left(\frac{(\sigma_1 - \sigma_2)^2}{\sigma_1 \sigma_2} \right) \times E^2 \sin 2\theta, \quad (2)$$

FIGURE 1: Scanning electron micrographs of BN nanosheets; (a) low magnification (x9000) and (b) high magnification (x12,000).

where V is the volume of a single graphite flake, θ is the angle between the electric field and the flake axis, ϵ_0 is the permittivity of free space, ϵ_2 is the relative dielectric constant of the resin matrix, and δ_1 and δ_2 are, respectively, the conductivities of the graphite flakes and the resin matrix [20, 23]. BN nanosheets require higher electric fields than GNs to control the anisotropy since BN is semiconducting. In particular, when nanosheets are oriented in a polymer and form a composite film with a thickness of the order of micrometers, the maximum electric field that can be applied is limited by the breakdown voltage of the polymer, which is lower than that of BN. The polysiloxane/BN composites produced in this study, which had thicknesses in the range 240–255 μm , were electrically insulating up to a voltage of 2.5 kV. The effect of DC electric fields on the anisotropic alignment of BN nanosheets in polysiloxane was compared using XRD analysis (Figure 2). The degree of anisotropy of the BN nanosheets perpendicular to the film plane was estimated by comparing the intensity ratios between c -axis ($I_{c\text{-axis}}$), ($2\theta = 26.76^\circ$) and a -axis ($I_{a\text{-axis}}$), ($2\theta = 41.60^\circ$)

$$\frac{I_{a\text{-axis}}}{I_{a\text{-axis}} + I_{c\text{-axis}}} \times 100(\%). \quad (3)$$

FIGURE 2: XRD patterns of polysiloxane/BN nanosheet composites produced by applying a DC electric field; (a) DC application (0 min), (b) DC application (10 min), (c) DC application (16 h).

The peaks at $2\theta = 26.76^\circ$ and 41.60° are due to diffraction from the (002) and (100) planes in BN, respectively. If all of the hexagonal BN nanosheets were oriented perpendicular to the normal to the film plane, the (002) BN intensity peak would be low and the (100) peak intensity would be high. When BN nanosheets were introduced into the polymer without applying an electric field, the peak intensity of the (002) plane of BN overwhelmed that of the (100) plane, as observed for the composite that was fabricated without applying an electric field (0 min). The (100) peak intensity increased greatly relative to the (002) peak intensity on application of a DC electric field. Applying an electric field for 10 min increased the intensity ratio of BN to 45.8%; the ratio increased to 52.8% on applying an electric field for 16 h. This indicates that the randomly distributed BN nanosheets were aligned perpendicular to the composite film plane on application of a DC electric field and this effect increases with the duration of the electric field.

Cross-sections of prepared polysiloxane/BN composites were cut and analyzed to investigate the distribution of BN nanosheets in the polymer matrix as function of the application of time of the electric field (Figure 3). The bright regions in the images indicate areas having high densities of BN nanosheets, whereas the darker regions indicate areas with very low densities of BN nanosheets. When agglomerations of BN nanosheets lie beneath the surface being observed, bright, clear images of individual nanosheets will be observed (see Figure 3(a)). When the microscope is focused on one agglomeration of nanosheets in the composite, nanosheets located at different depths will appear bright, but blurred. The BN nanosheets in the composite prepared without applying an electric field have a homogeneous distribution. However, when an electric field

FIGURE 3: Cross-section view of polysiloxane/BN nanosheet composite film by digital microscope; (a) DC application (0 min), (b) DC application (10 min), (c) DC application (16 h).

is applied, the nanosheets moved to the side of the surface where the positive electrode was located. Both Figures 3(b) and 3(c) show higher BN nanosheet densities on the left-hand side, where the positive electrode was located. The BN nanosheet density on the left-hand side increased with longer application of the electric field (Figure 3(c)). In addition, the LABNs form filament-like structures inside the polymer; these structures are also aligned perpendicular to the film plane. Figure 3(b), which shows the sample prepared by applying an electric field for 10 min, reveals that the LABN

tips are bent and that the BN density on the left-hand side (i.e., near the negative electrode), is considerably higher than that for the composite prepared by applying an electric field for 16 h. This indicates that the BN nanosheets move from left to the right by electrophoresis and that this movement had not finished after 10 min. This means that the filament structures of LABNs were formed during application of an electric field for 10 min and were partially destroyed when the electric field was removed because of insufficient cross-linking of the prepolymer. Estimating from the high

FIGURE 4: Cross-sectional SEM views of polysiloxane/BN composite film; (a) without and (b) with the application of DC electric field.

FIGURE 5: Comparison of thermo conductivities of polysiloxane/BN composite film.

intensity ratio of BN nanosheets in the polymer (Figure 2), the BN nanosheets within the filament structures of LABNs are aligned perpendicular to the film plane with a high anisotropy.

The alignment and distribution of the BN nanosheets in the LABNs are shown in cross-sectional SEM images (Figure 4). In the composite prepared without applying an electric field (Figure 4(a)), the BN nanosheets are randomly distributed with a low density. In contrast, the composite prepared by applying a DC electric field for 16 h have a comparatively high BN density and the majority of nanosheets are longitudinally aligned perpendicular to the film plane. Even though the BN nanosheets are tilted in different directions, their longitudinal surfaces are aligned perpendicular with the film plane. It is probable that the BN nanosheets become charged due to the polarization induced by the high DC electric field (2.5 kV). Carbon nanotubes have a high dipole moment along their longitudinal axis,

which is aligned in the direction of electric field [21]. The charge density has been predicted to increase at the edges of single-walled carbon nanotubes (SWCNTs) [13]. Under a high DC electric field, the surfaces of BN nanosheets are polarized stronger at their edges of their longitudinal surfaces and consequently they align parallel to electric flux. Coulombic attraction is another force that acts between the oppositely charged ends of nanotubes [24], and it causes the formation of linear structures at both ends of each nanotube; the nanotubes form very dense groups in solution due to electrophoresis. Linear bundles of SWCNTs have been fabricated in an electric field, but the SWCNT surfaces were modified by tetraoctylammonium ions and the experiment was performed in tetrahydrofuran [17]. The fabrication of LABNs in a polymer in the present study is significant because elongated structures with linearly aligned nanosheets were formed in a polymer resin by applying a high electric field without modifying the BN nanosheet surface.

The thermal conductivities of the prepared composites were measured to investigate the effect of anisotropy and the formation of LABNs on the thermal conductivity. Figure 5 shows a plot of the thermal conductivity as a function of the diffraction intensity ratio of BN nanosheets (5 vol% in the polymer) in the composites. The polysiloxane prepared by cross-linking the prepolymer mixture without adding BN nanosheets had a thermal conductivity of $9.3 \times 10^{-2} \text{ W/m}\cdot\text{K}$; this was remarkably increased by adding 5 vol% BN nanosheets. Furthermore, the thermal conductivity increased with increasing BN intensity ratio, which confirms that the thermal conductivity of polysiloxane/BN nanosheet composites increased as the anisotropic alignment of BN nanosheets in the polymer matrix increases.

3.2. Fabrication Mechanism of LABNs. The formation of filaments of LABNs contributed to the enhanced thermal conductivity, as shown in Figure 6. As mentioned above, nanosheets can be polarized by a strong DC electric field and the charge density increases toward the longitudinal

FIGURE 6: Schematic illustration of formation of LABNs under a DC electric field (2.5 kV); (a) rotation, (b) electrophoresis, (c) LABNs bundles.

FIGURE 7: Schematic illustration of heat diffusion route in polysiloxane/BN nanosheets composite film; (a) random distribution, (b) ordered distribution with LABNs.

edges [13, 24]. The nanosheets align themselves parallel to the electric field to minimize the electrostatic energy and to overcome the free energy of the system, forming a stable configuration [1, 15]. The edges of BN two nanosheets in close proximity will become attached to each other by Coulomb attraction during electrophoretic movement in the prepolymer mixture before it is completely cured. Based on Figures 3 and 4, the filaments of LABNs are considered to be composed of groups of linearly aligned nanosheets. Some groups are connected while others are separate; they form a linear assembly as one component of the filament structure shown in Figure 6. These LABN structures enhance the thermal conductivity since heat diffuses along the path formed by linearly aligned BN nanosheets and by condensing the heat diffusion through the polymer (Figure 7). This result may be explained by the different thermal conductivities of polysiloxane and BN, and of the c - ($\perp$) and a -axis ($\parallel$) of BN nanosheets. The thermal conductivity increases remarkably on the addition of BN nanosheets to the polymer (see Figure 5) and the thermal conductivity of BN nanosheets parallel to the a -axis ($\parallel$) is 20 times higher than that parallel to the c -axis ($\perp$) [10, 11]. Consequently, the fabricated LABNs bundles in the polysiloxane/BN nanosheet composite films have the potential to be used in semiconductor applications that require electric insulators with high thermal conductivities.

4. Conclusions

Linear assemblies of BN nanosheets (LABNs) were successfully fabricated within polysiloxane/BN nanosheet composite film under a high DC electric field (2.5 kV). The filament structures of LABNs were composed of groups of linearly aligned BN nanosheets, which were fabricated by the mixed effects of polarization, dipole-dipole moment, electrophoresis, and coulombic attraction. The BN nanosheets which make up the LABNs showed high anisotropy, and their linear attachment was regarded to be critical for the higher thermal conductivity of the polysiloxane/BN composite film. The fabricated LABNs were considered to motivate efficient thermal conduction by transferring heat through a series of BN nanosheets which were aligned perpendicular to the composite film plane and by avoiding conduction through the polymer. This paper introduces filament structured linear assemblies of nanosheets fabricated within organic/inorganic nanocomposite material. Further works will focus on the fabrication of linear bundle bridges of BN nanosheets by alternating the DC electric field direction to enhance thermal conductivity.

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