Polycarbonate–hexagonal boron nitride composites with better dielectric properties for electronic applications

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Abstract

Hexagonal boron nitride (h-BN) reinforced polycarbonate (PC) composites were fabricated using solution method followed by hot pressing. The content of h-BN was varied from 0 to 60 wt% (~44.4 vol%). The morphology, electrical volume resistivity, microhardness, and dielectric properties of the composites were characterized. Scanning electron microscopy showed uniform dispersion of h-BN particles in the PC matrix. X-ray diffraction and Fourier transforms infrared spectroscopy showed no change in morphology of PC present in the composites. The dielectric constant of the composites containing 60 wt% h-BN increased to 3.9. The dissipation factor of the composites reduced to less than 0.007 and the dispersion in dielectric constant of the composites became stable over the frequency from 1 kHz to 10 MHz compared to that of pure PC. The electrical volume resistivity of the composites remained close to that of pure PC. The microhardness increased approximately 25% at 50 wt% h-BN content and thereafter, a slight decrease in microhardness was observed.

Keywords Electrical properties · Hardness · Morphology · Polymer matrix composites

1 Introduction

Polymer matrix composites reinforced with thermally conducting but electrically insulating ceramic particles are widely used for the applications in printed circuit boards (PCBs) and electronic packaging substrates because of their high electrical resistivity, flexibility and low processing temperatures. Compared to metals and ceramics, pure polymers exhibit high coefficient of thermal expansion (CTE), low thermal conductivity and mechanical properties. Therefore, these drawbacks have been minimized by reinforcing electrical insulating but thermally conducting ceramics such as alumina, boron nitride (BN), aluminum nitride, silica, and silicon nitride into the polymers [1, 2]. In details to know, readers can refer a good review on polymer–ceramic composites recently published by Sebastian and Jantunen [3]. Among above mentioned ceramics, hexagonal-BN (h-BN) is widely used by the researchers

due to its low CTE, excellent thermal conductivity, good dielectric and mechanical properties. Its high electrical resistivity makes it an interesting reinforcement to achieve outstanding composites [4–14]. Additionally, the h-BN is comparatively softer and easily processable. The through-plane thermal conductivity is about two order of magnitude less than that of in-plane thermal conductivity of h-BN. The actual values of thermal conductivity of a material depends upon the measurement direction, temperature and defects present in the sample [15–17]. In contrast, other ceramics are abrasive in nature and can possibly damage the processing equipment such as extrusion and molding machines during processing. The addition of 5 wt% h-BN powder increased the thermal conductivity of the epoxy composites approximately two-fold. Its layered structure has been attributed as one of the excellent reason for the significant increase in thermal conductivity [18]. Similarly, the thermal conductivity of the cured

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35 wt% h-BN filled 2,2'-diallylbisphenol A (DBA)-modified 4,4'-bismaleimidodiphenylmethane (BDM) composites was increased to about 3.3 times of that of pure resin. Moreover, the linear CTE of this composite was decreased significantly [19–21]. Despite such improvements in properties, the effect of h-BN on dielectric and mechanical properties of polycarbonate (PC) is hardly studied, so far. PC is a thermoplastic polymer which is known to have amorphous structure and good transparency. Compared to epoxy, it exhibits excellent fracture toughness, low water absorption, high volume resistivity and dielectric strength. Further, the addition of suitable type, size and volume fraction of reinforcements modifies its electrical, mechanical and optical properties [22–28].

In view of above, the aim of this study is to explore the effect of 0–60 wt% (or 0–44.4 vol%) h-BN particles on the microhardness, dielectric properties and volume resistivity of the PC/h-BN composites prepared by solution method followed by hot pressing. The prepared composites were characterized by X-ray diffractometer (XRD), scanning electron microscope (SEM), Fourier transforms infrared spectrometer (FTIR), Vickers hardness tester, precision impedance analyzer, and electrometer to get the

information about morphology/structure, mechanical and electrical properties of the PC/h-BN composites for the applications in PCBs/electronic substrates.

2 Experimental

2.1 Materials

The PC granules and h-BN (CAS Number: 10043-11-5) powder were obtained from the local market and M/s Sigma Aldrich Chemicals, respectively. According to manufacturer the size and purity of h-BN were about 1 μm and 98%, respectively. The h-BN was used in as received condition as reinforcement. Tetrahydrofuran (THF) was used to dissolve the PC. Figure 1 shows the SEM of pure h-BN powder at various magnifications. It can be seen that the h-BN particles have platelet-like morphology. The width and thickness of the platelets are about 5–10 μm and < 0.5 μm , respectively, indicating its aspect ratio more than 10.

Fig. 1 SEM of h-BN powder at a magnifications of **a** 500 $\times$, **b** 2000 $\times$ and **c** 5000 $\times$

2.2 Fabrication

The PC/h-BN composites were fabricated using solution-method followed by hot pressing. The h-BN powder was suspended in THF using ultrasonic bath. Similarly, PC granules were dissolved separately in THF. Both solutions were sonicated for about 45 min and then, mixed together. The mixture was further sonicated for 15 min. This was done to achieve uniform distribution of h-BN particles in the PC matrix. Then the composite film of uniform thickness was casted by pouring solution onto a cleaned glass plate. The film was dried in a vacuum oven at 150 °C for 2 h. The films obtained were cut into fine pieces, weighed and hot-pressed at 230 °C under a pressure of 45 MPa. A cylindrical samples (or pellets) with 13 mm in diameter and 1 mm in thickness were obtained using the method as reported in our earlier paper [19]. The h-BN content was 30, 50 and 60 wt% which corresponds to vol% of 18.6, 34.8 and 44.44, respectively. The vol% of h-BN was calculated using the equation: $V_f = W_f / [W_f + W_m (\rho_f / \rho_m)]$, where, W_f is the weight fraction, and ρ_f is the density of h-BN. The volume fraction of h-BN can be calculated by dividing vol% by 100, i.e., 18.6 vol% is equivalent to 0.186 volume fraction. W_m and ρ_m are the weight fraction and density of the PC matrix, respectively.

2.3 Characterization

Theoretical density of the composites was calculated by the rule of mixtures (RoM) using the density of h-BN 2.25 g/cm³ and of PC 1.2 g/cm³. For theoretical density, it was assumed that there were no voids and no loss of h-BN or PC during processing. Experimental density of the composites was determined by the Archimedes Principle [19]. The crystal structure of h-BN particles and morphology of PC were determined by a Philips X'Pert PANalytical PW 3040/60 X-ray diffractometer. XRD patterns of pure PC pellet, h-BN powder and composite containing 30 wt% (18.6 vol%) h-BN were recorded using Ni filtered Cu K α radiation ($\lambda = 1.54$) generated at 40 kV and 30 mA. Scanning electron microscope (SEM) (JOEL, JSM-6360A) was used to examine morphology and dispersion of h-BN particles in the PC matrix. The composites were fractured in liquid nitrogen and then, polished using various emery papers followed by lapping. Before SEM examination, the polished surfaces of samples were coated by gold–palladium alloy to avoid charging using Sputter. Fourier transforms infrared (FTIR) spectra were recorded by a JASCO FTIR-6100 spectrometer in the range of 400–4000 cm⁻¹. Thin samples of 0.2 mm thickness were prepared for this analysis. KBr powder was mixed with h-BN powder to get transparent pellet for examining in transmission mode FTIR. The polymer films or h-BN/KBr pellets were placed

under IR lamp for 2–3 min to remove moisture. Then the samples were placed on the Aluminum sample holder for actual analysis. The dielectric constant or permittivity was obtained from the measurement of capacitance using precision impedance analyzer [Wayne Kerr Electronics, 6515B, UK] at frequencies varying from 10 kHz to 15 MHz at 30 °C. The dielectric constant (ϵ) was obtained from the measurement of capacitance (C) using equation; $\epsilon = C.t / (\epsilon_0.A)$, where, ϵ_0 (8.854×10^{-12} F/m) is the permittivity of free space, t is the thickness and A is the cross sectional area of the sample. The dielectric loss tangent or dissipation factor which is the ratio of dielectric loss to dielectric constant was obtained directly from the instrument. For the measurement of electrical volume resistivity, samples were coated with a thin coating of silver paste to minimize contact resistance. The resistance of samples was measured using electrometer (Keithley 6517B, UK) at an applied voltage of 250 V to the test samples. Then, the volume resistivity (ρ) was calculated by the relation $\rho = R.(A/t)$, where R is resistance of the sample. Microhardness was determined using Vickers microhardness tester (FM 700 Type D, Future Tech, Japan). The average of six readings was considered as the final microhardness.

3 Results and discussion

3.1 Density

Figure 2 shows experimental density and theoretical density of the composites as a function of h-BN content. As the h-BN content increases the density of composites increases linearly. The increase in density may be attributed to the higher density of h-BN (2.25 g/cc) than that of pure PC matrix (1.20 g/cc). It can also be seen that the

Fig. 2 Theoretical and experimental densities of the PC/h-BN composites

experimental density is very close to that of theoretical density indicating almost porosity free samples. The resultant sound samples are probably due to good processing conditions which resulted in good adhesion between the h-BN particles and the matrix.

3.2 X-ray diffraction (XRD)

Figure 3 shows the XRD pattern of pure PC sample, h-BN powder and PC/h-BN composite containing 30 wt% (or 18.6 vol%) h-BN. The diffraction peaks of h-BN powder correspond to (002), (100), (101), (102), (004) and (110) planes of the h-BN, respectively, with lattice parameters of $a=0.2504$ nm and $c=0.6656$ nm (JCPDS ICSD 34-0421) [29, 30]. This shows that h-BN has hexagonal structure. A diffraction peak at about $2\theta=43.3^\circ$ (d-spacing=0.2089 nm) corresponding to cubic-BN was not found indicating that h-BN does not contain cubic-BN trace. Pure PC exhibits a broad halo at about $2\theta=17.6^\circ$ indicating that it does not have long range ordering of polymer chains. Moreover, composite does not show any new diffraction peak other than the diffraction peaks of pure constituents of h-BN and PC. This shows that PC matrix is not influenced by the h-BN loading.

3.3 FTIR analysis

Fourier transforms infrared (FTIR) spectra depict functional groups and fingerprint regions for the materials. Figure 4 shows the FTIR spectra of pure PC, h-BN powder and the composite. Two characteristic absorption bands at about 808 cm^{-1} and 1363 cm^{-1} are observed for the h-BN which can be attributed to the B–N bending absorption and

Fig. 3 X-ray diffraction patterns of pure PC, h-BN powder and PC/h-BN composite

Fig. 4 FTIR spectra of pure PC, h-BN powder and PC/h-BN composite

B–N stretching, respectively. An absence of characteristic absorption band at 1100 cm^{-1} indicates that h-BN powder is free from cubic-BN [29–32]. The strong characteristic absorption peak of raw h-BN at 1376 cm^{-1} resulted from the in-plane B–N stretching vibration. The peak around 819 cm^{-1} was the B–N–B out-of-plane bending vibration and the broad band at 3442 cm^{-1} was for the hydroxyl groups and amino groups at the edge planes of BN or the surface moisture [30]. The characteristic infrared band of carbonate linker/carbonyl functional group (C=O) is at 1802 cm^{-1} . The stretching of the C–C bond from the phenyl group (benzene ring) occurs at $\approx 1601\text{ cm}^{-1}$ and the C=C bond vibration at 1496 cm^{-1} . The stretching of the ester group (O–C–O) occurs around 1154 cm^{-1} . It is interesting to see that the carbonyl band (C=O) and the C=C band of PC fraction of the 18.6 vol% BN filled composite film is shifted from 1802 to 1786 cm^{-1} and from 1541 to 1513 cm^{-1} , respectively. The changes in the observed shift may be attributed to good physical interaction between the h-BN particles and the PC molecules of the composites after h-BN loading [32].

3.4 Scanning electron microscopy (SEM)

Figure 5a, b shows SEM images of the polished (but without etched) composite samples at a magnification $2000\times$. The h-BN particles are almost uniformly distributed and as h-BN content increases particle to particle contact increases and microstructure becomes more and more dense. Hardly, there are cracks or pores visible on the surface of composites. Due to lower atomic number of h-BN, the visibility of h-BN particles is not good, hence etching of the composite containing 50 wt% (34.8 vol%) h-BN sample was carried out using a 2% w/v solution of potassium

Fig. 5 SEM of the unetched composites containing **a** 34.8 vol%, **b** 44.44 vol% h-BN powder, and of **c** 34.8 vol% h-BN composites after etching at a magnification of 2000 $\times$

permanganate in a mixture of 4 volumes of orthophosphoric acid and 1 volume of water. The polished cross-sections of the samples were etched for 2–3 min [20] and then observed under SEM. Figure 5c shows SEM of etched composite. It can be clearly seen that h-BN particles are uniformly dispersed in the matrix and h-BN particles are touching each other in the matrix. This is due to the higher aspect ratio of h-BN plates as observed in Fig. 1. This may be beneficial from the advantage of the thermal management where heat dissipation is the main requirement. In addition, most h-BN particles tightly embedded within the matrix, implying the presence of strong interface interaction between h-BN particle and the matrix. A strong filler-matrix interfacial adhesion is expected to improve the properties of the final PC/h-BN composites.

3.5 Electrical properties

It is well known that the speed of signal is inversely proportional to square root of dielectric constant. So in general, lower dielectric constant is preferred for the PCBs as it has lower impedance on signal propagation. Figure 6a, b shows the effect of h-BN content on the dielectric

constant and dissipation factor, respectively, as a function of frequency. It can be seen that the dielectric constant of pure PC is about 2.9 (at 1 MHz) while it varies between 3.9 and 4.1 for the pure BN depending upon frequency [6]. As the frequency increases dielectric constant of pure PC decreases. However, the dispersion (i.e., decreasing trend) in dielectric constant of the composites is almost insignificant. The highest value of dielectric constant of the composites achieved is below 3.8. The higher dielectric constants for the composites compared to pure PC is due to the higher dielectric constant of h-BN compared to pure matrix. The decrease in dispersion in the dielectric constant of composites compared to pure matrix can be attributed to the better interaction between the h-BN particles and the matrix as confirmed by FTIR and SEM. Similar to the dielectric constant, a lower dissipation factor is preferred. From Fig. 6b, it can be seen that the dissipation factor (at a constant frequency) decreases with increase wt% of h-BN. This is due to the fact that h-BN has dissipation factor as low as 0.0002 in comparison with pure PC which is about 0.0081 [33]. Moreover, better interaction between the h-BN particles and the matrix decreases mobility of the polymer chains, thus reduces frictional heat (or dissipation

Fig. 6 **a** Dielectric constant versus frequency, and **b** dissipation factor versus vol% of h-BN of the PC/BN composites

factor). This certainly helps to lower the dissipation factor of the composites. For example, the dissipation factor of 44.4 vol% h-BN filled composite is reduced to below 0.007. A low and stable dielectric loss is beneficial in reducing power loss and heat production, which, in turn, results in increased operating speeds and reduced crosstalk [26].

The values of electrical volume resistivity for the pure PC is found about $3.4 \times 10^{12} \Omega \text{ cm}$ which is gradually increased with increasing h-BN content. The volume resistivity of 44.4 vol% h-BN composite is about $9.2 \times 10^{12} \Omega \text{ cm}$, i.e., there is no significant change in resistivity of the composites as compared to pure PC. For thermal interface materials, light emitting diodes, thermally conductive paste, PCBs and/or microelectronic packaging, the electrical insulation property is a key factor to ensure the security and stability of the electronic components when they operate under electric field. The PC/h-BN composites retained good electrical insulation properties, because of the inherent electrical insulating characteristic of the h-BN powder and PC matrix. These PC/h-BN composites possessed desirable properties for the PCBs that requires low dissipation factor, low dielectric constant and high electrical insulation

Fig. 7 Microhardness of the PC/h-BN composites

[30, 34]. It has been studied that the PC/h-BN composites not only exhibit significant increase in hardness, decrease in dissipation factor but also maintain almost the same volume electrical resistivity as the pure PC. This originates from the electrically insulating property of the h-BN and better interaction between h-BN and the matrix. Therefore, the present PC/h-BN composites hold great promise as an alternative PCB material.

3.6 Microhardness

Figure 7 shows microhardness of the composite as a function of h-BN content. The microhardness of the composites increases gradually with increasing h-BN particles up to 34.8 vol% h-BN content, i.e., the microhardness increased from 19.84 kg/mm² (for pure PC) to 24.84 kg/mm² for 34.8 vol% composite indicating an increase of about 25% compared to pure matrix. The increase in microhardness might be attributed to comparatively higher hardness of h-BN and its better dispersion in the matrix which resists the deformation of the composites. Thereafter, a slight decrease in microhardness was found. Probably it is due to the formation of aggregates at higher loading. Similar trend was found for the polyurethane/BN nanotubes composites [35].

4 Conclusions

For the first time, polycarbonate/h-BN composites were fabricated using solution method followed by hot pressing at 230 °C and 45 MPa. The experimental density of the composites was found close to the theoretical density indicating pores free samples. XRD did not show change in morphology of the PC. SEM revealed uniform dispersion

and distribution of the h-BN particles in the matrix. FTIR and SEM indicated better interaction between the h-BN particles and the matrix. The dielectric constant of pure PC is 2.9 (at 1 MHz) which was increased marginally to about 3.9 for 44.4 vol% composites. Interestingly, the dispersion in dielectric constant of the composites with increasing frequency was reduced significantly and the dissipation factor of 44.4 vol% h-BN filled composite was reduced to below 0.007. The microhardness of the composites improved approximately 25%. In brief, the PC/h-BN composites not only exhibit significant increase in hardness, decrease in dissipation factor but also maintain almost the same volume electrical resistivity as the pure PC. This originates from the electrically insulating property of the h-BN and better interaction between h-BN particles and the matrix. Therefore, the present PC/h-BN composites hold great promise as an alternative PCB material.

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Compliance with ethical standards

Conflict of interest On behalf of all authors, RKG (corresponding author) states that there is no conflict of interest.

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