Effective Surface Treatments for Enhancing the Thermal Conductivity of BN-Filled Epoxy Composite

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ABSTRACT: To improve the thermal conductivity of BN-filled epoxy composite, admicellar polymerization was used to coat polystyrene and polymethyl methacrylate on the BN surface to improve the interfacial adhesion in the composite. The treated surface was characterized by FTIR and contact angle measurements. The results show that the admicellar treatment led to improved wettability of epoxy resin on the treated surface. Thermal conductivity of the composite increased from 1.5 W/mK for untreated BN to 2.69 W/mK when the admicellar-treated BN was used, indicating improvement in the interfacial adhesion between BN and epoxy resin in the composite. The mechanical properties of the composite also improved significantly. The surfactant : monomer molar ratio of 1 : 10 was found to be the optimum condition for the admicel-

lar polymerization process. The solubility parameter concept was used to explain the difference in the effectiveness of polystyrene and polymethyl methacrylate. When compared to the more conventional silane treatment, admicellar treatment was found to be more effective in improving the interfacial adhesion between the BN particles and epoxy resin. SEM micrographs of the fractured surface of the composite further confirm the improvement in the interfacial adhesion after the admicellar treatment. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 119: 3234–3243, 2011

Key words: thermal conductive composite; epoxy composite; boron nitride; surface modification; admicellar polymerization; silane treatment; interfacial adhesion

INTRODUCTION

Hexagonal boron nitride (h-BN) is known to be a nonmetallic filler with a high intrinsic thermal conductivity, high temperature resistance, and excellent oxidation resistance. It is therefore an attractive choice for use as a filler to enhance the thermal conductivity of a composite and there have been numerous studies on the properties of BN-filled composites.[1–4] The favorable property of BN is attributed to its nonspherical (platelet) shape and high thermal conductivity in the basal plane.[5] Hill and Supancic[6] also studied platelet-shaped particles of similar size and shape. They found that flat platelets provide higher heat transfer than predicted value because of their high contacting surface area when the BN plates stack upon one another. The main challenge with BN is that the surface is very inert. This leads to poor interfacial adhesion between BN and the matrix in a composite.[7]

It is known that the transport of heat in nonmetals occurs through lattice vibrations in the solid.[8] An increase in vibrational energy in one part of the solid, associated with an increase in temperature, will be transmitted to an adjacent area along the path of the temperature gradient. Hence, in a composite, the efficiency in heat transfer depends greatly on the contact point between the filler and matrix.[9] A good contact between the two phases is critical to the efficiency of heat flow.[10] It has been shown that surface treatments of the filler to improve the interfacial adhesion between the filler and the polymer matrix, e.g., by silane treatment, significantly improve the heat transfer capability of the composite.[11,12] One other method that can be used to improve the adhesion between the filler and the matrix is to coat the filler surface with a thin layer of polymer that is more compatible with the matrix. The coating can be done via a process called admicellar polymerization in which the polymerization takes place in a surfactant bilayer adsorbed on the substrate surface.[13] The resulting polymer layer formed on the substrate surface has been found to be ultrathin, in nanoscale, and very uniform.[14,15] The process has been successfully used to coat a thin polymeric layer on a wide range of inorganic substrates including glass fibers,[16] silica,[17] and alumina.[18–20] It has also been used to improve the adhesion between the filler and matrix to improve the mechanical strength of the composites.[21,22] However, the application of the process to improve the heat

transfer of a particulate-filled composite has not been studied. In the present work admicellar polymerization was used to coat two different polymers, polystyrene (PS) and polymethyl methacrylate (PMMA), on the BN particles to improve its interfacial adhesion with the epoxy matrix. The effect of the treatment on the thermal conductivity of the composite was studied and the results were also compared with the more conventional silane treatments. The effect on the mechanical properties of the composites was also determined.

EXPERIMENTAL

Materials

The epoxy resin bisphenol-A-epichlorohydrin (EPO-TEC YD128) and the curing agent cycloaliphatic amine (TH730) were obtained from Aditya Birla Chemicals (Thailand) Ltd. The hexagonal BN (PCTL30MHF) with an average size of 30 μm was supplied by Saint-Gobain Ceramics and Plastics, USA. The silane coupling agents γ-glycidoxypropyl-trimethoxy silane (GPS) and 3-aminopropyl triethoxy silane (APS) were supplied by Sigma-Aldrich (United States). Hexadecyl pyridinium chloride (HDPyCl, 98% purity), methyl methacrylate (MMA, 99% purity), styrene (99% purity), and potassium persulfate ($K_2S_2O_8$, 98% purity) were purchased from Fluka (Switzerland). Ethanol and sodium hydroxide were purchased from Merck (Germany). Methyl methacrylate and styrene were washed with 1% aq. NaOH to eliminate inhibitor prior to use.

Preparation of the admicellar-treated BN

Admicellar polymerization of each monomer (MMA and styrene) was carried out in two steps. First, 5 g of BN particles was added to 100 mL of an aqueous solution containing 1.0 mM HDPyCl surfactant, which is the concentration just above the CMC of the surfactant[23] to ensure maximum surfactant adsorption with minimum emulsion polymerization in the aqueous phase. Monomer was then added to give a surfactant : monomer molar ratio of 1 : 2.5, 1 : 5, 1 : 7.5, 1 : 10, 1 : 12.5, 1 : 15, to cover the range below and above the optimum ratio found by other workers.[24,25] The initiator $K_2S_2O_8$ was then added to give a monomer : initiator molar ratio of 1 : 10.[26] The mixture was shaken in a shaker bath at 30°C for 24 h to allow for the adsorption of surfactant onto the BN surface and the adsolubilization of the monomer into the surfactant bilayer. In the second step, the temperature was raised to 70°C to initiate the polymerization reaction which was allowed to take place for 24 h. At the end of the reaction time, the treated BN was washed several times with a mixture of 70/30 (v/v) water/ethanol at room temperature to remove remaining monomer and the upper-layer surfactant of the surfactant bilayer to expose the coated polymer on the BN surface. The sample was finally dried in the oven at 50°C for 24 h.

Preparation of silane-treated BN

The calculated amount of silane coupling agent was added into an aqueous solution at pH 4.5 to give a concentration of 0.025, 0.05, 0.075, 0.10, and 0.15 wt %, respectively. Then, 20 g of BN particles was treated in 100 mL of the prepared silane solution for 2 h at room temperature. The BN particles were then filtered and dried in the oven at 50°C for 24 h.

Preparation of the BN-epoxy composite

The BN particles were first dried in a vacuum oven at 80°C for 24 h prior to processing. The calculated amount of BN to give 33 vol % was added gradually to 60 mL of epoxy resin contained in a 250 mL plastic beaker with continuous stirring. The mixture was then stirred by a mechanical stirrer (A.L.C. International S.r.l. Class I, Italy) at 300 rpm, 30 min. Next, 36 mL of curing agent was added into the mixture which was then stirred until homogeneous. The mixture was then poured into a stainless steel mold 14 × 20 × 0.3 cm³ in dimension. The open mold was then placed in a vacuum oven at 50°C for 10 min to evacuate the entrapped air. The mold was then closed by a 26 × 26 cm² metal plate and placed in the compression molding machine under 15 tons of loading for curing at 80°C for 1 h.

Characterization of admicellar-treated BN surface

The untreated BN and admicellar-treated BN particles were prepared in the form of KBr pellets for the Fourier transform infrared (FTIR) measurements on a Nicolet 560 FTIR spectrometer (United States). Subtractive FTIR spectra of the treated samples were obtained using the untreated BN as the base line. The Field Emission Scanning Electron Microscope (FE-SEM), Hitachi *S*-4800 (Japan), was used to study surface morphology of the untreated and admicellar-treated BN particles, as well as the fractured surface of the composites.

Contact angle and wettability of surface modified BN particles

The Drop Shape Analyzer (KRUSS, DSA 1 V 1.80, Germany) was used to measure the contact angles of water and epoxy resin on either admicellar- or silane-treated BN surface. BN particles, weighing 1.5 g, were compressed using a hydraulic pellet press at
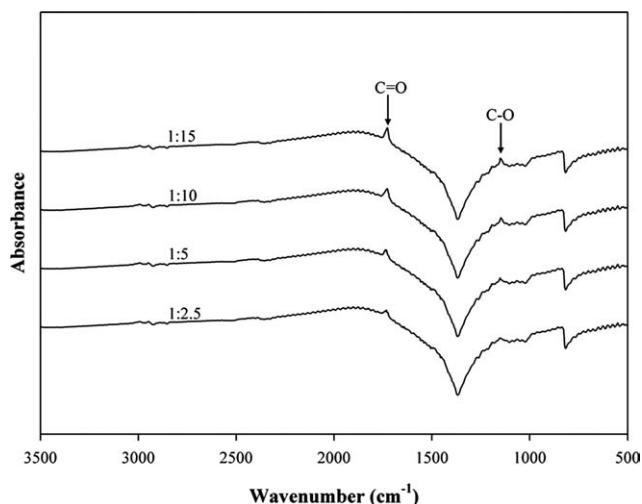
Figure 1 FTIR spectra of PMMA-coated BN with varying surfactant : monomer molar ratio.

the pressure of 7 tons for 1 min to obtain a BN disc 1 cm in diameter and 3 mm in thickness. Either 20 μL of water or 20 μL epoxy resin droplet was placed onto the surface of the BN disc using 100 μL microsyringe and the contact angle of the droplet was measured. The test was performed at room temperature. Three readings were taken from each disc and each result was the average of the readings from two discs.

Measurement of the thermal conductivity of the composite

Thermal conductivity of the composites was measured using a Hot Disk Thermal Analyzer (Hot Disk AB, Uppsala. Sweden). A minimum of three individual measurements was performed for each specimen ($20 \times 20 \times 3$ mm^3) with the sensor (3-mm diameter) placed between two similar slabs of material. The sensor supplied a heat-pulse of 0.03 W for 15–20 s to the sample and the associated change in temperature was recorded.

Determination of mechanical properties

Flexural testing was performed based on ASTM D 790-98 using the three-point bending tests. The testing was conducted by using Instron series IX Automated Materials Testing System model 3366 with load cell of 10 kN. The crosshead displacement rate was set at 5 mm min^{-1}. The specimens used for flexural testing were bars of rectangular cross section (flatwise) with a length of 60 mm and width of 14 mm. Three specimens were tested for each sample and the average value was reported. Impact strength was determined using a Pendulum Impact Tester (Zwick, Germany) and the testing method was in ac-

cordance with ASTM D-256. For each sample, five specimens measuring $63.5 \times 12.7 \times 3$ mm^3 were tested and the average value was reported.

RESULTS AND DISCUSSION

Characterization of admicellar-treated BN particles

In this study methyl methacrylate and styrene monomers were used to prepare admicellar-treated BN surface. Figures 1 and 2 show the FTIR spectra of PMMA- and PS-coated BN particles at varying surfactant : monomer molar ratio, respectively. From Figure 1, the characteristic absorption bands of PMMA at 1732 and 1243 cm^{-1}, assigned to C=O stretching and C—O stretching,[27] can be seen in MMA-treated BN. In Figure 2 the characteristic peaks of PS at 1601, 1495, and 1451 cm^{-1} assigned to aromatic C=C stretching, and at 701 cm^{-1} assigned to out-of-plane aromatic C—H bending can be seen.[28] The results confirm that PMMA and PS were successfully coated on BN particles by admicellar polymerization and the intensity of the absorption peaks were found to increase with increase in the amount of monomer used.

SEM micrographs of untreated and admicellar-treated BN surfaces are shown in Figure 3. The 1 : 10 PMMA- and PS-coated surfaces shown in Figure 3(c,d) can be seen to be quite different from the clean surface of unmodified BN in Figure 3(a,b). The micrographs show clear evidence of deposition of relatively uniform layer of polymer on the treated surface. Through our observation at various magnifications, we found that at magnifications below 8000×, the treated surface was hardly distinguishable from the unmodified surface, indicating that the coated polymer layer must be quite thin and
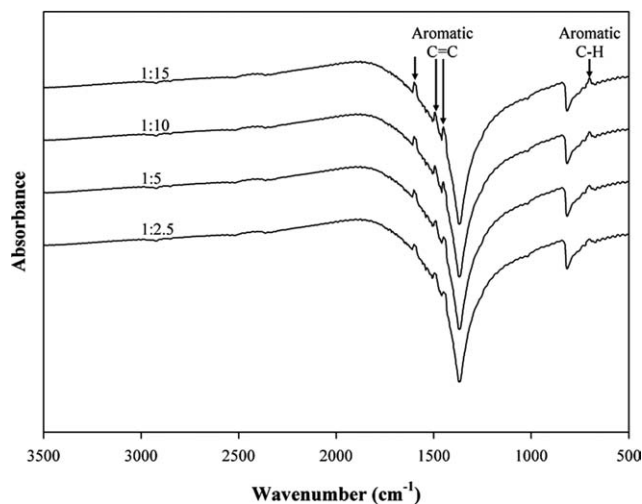

Figure 2 FTIR spectra of PS-coated BN with varying surfactant : monomer molar ratio.
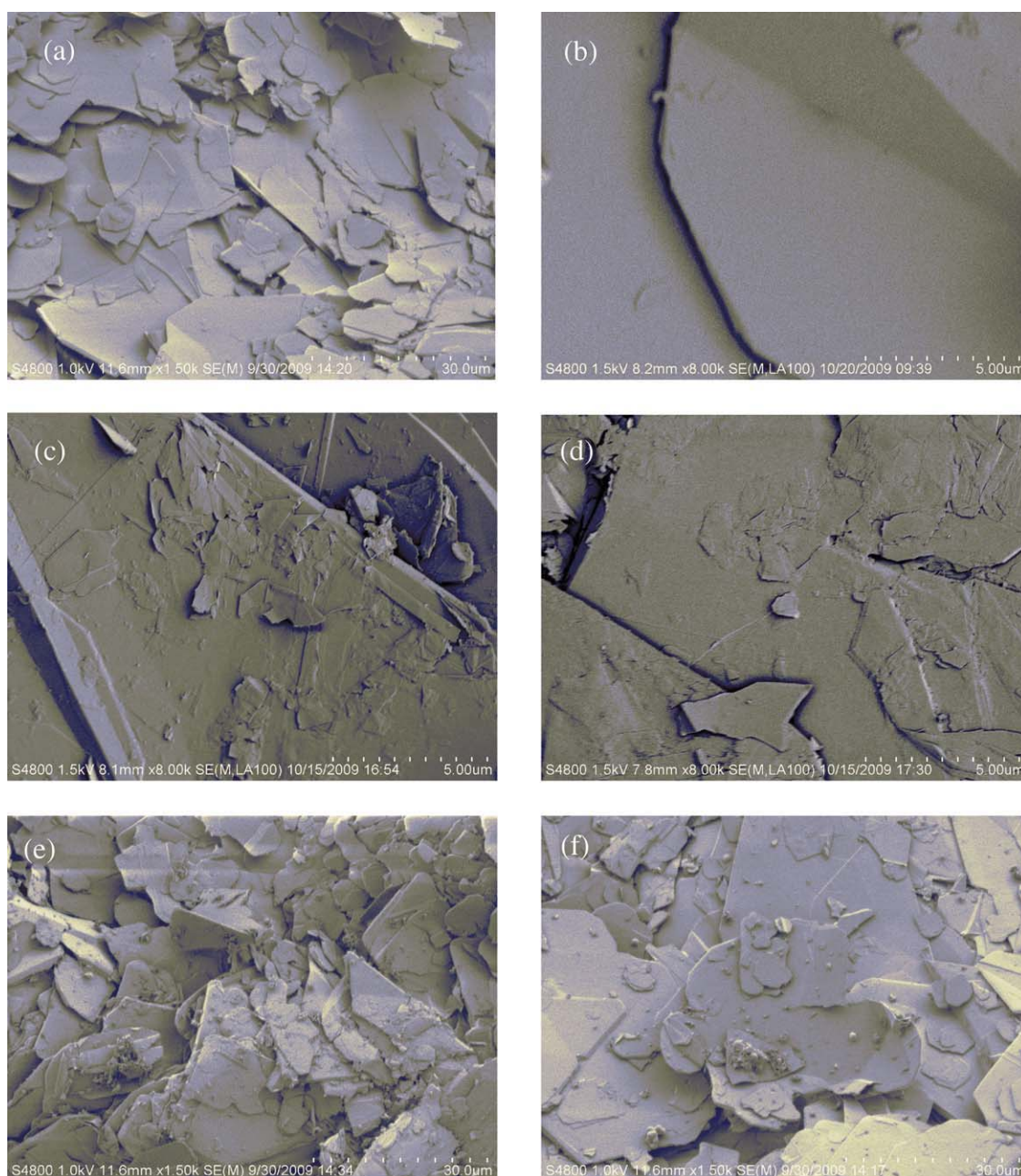
Figure 3 SEM micrographs of (a) untreated BN particles (×1500), (b) untreated BN particles (×8000), (c) 1 : 10 PMMA-coated BN particles (×8000), (d) 1 : 10 PS-coated BN particles (×8000), (e) 1 : 15 PMMA-coated BN particles (×1500), and (f) 1 : 15 PS-coated BN particles (×1500). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

uniform. When the surfactant : monomer ratio was increased to 1 : 15, there was deposition of polymer particles on the BN surface as shown in Figure 3(e,f), indicating that polymerization had taken place in the aqueous phase with the resulting polymer particles depositing on the BN surface. Hence, in surface coating via admicellar polymerization, there is an optimum monomer concentration which will ensure maximum surface coating with no unwanted polymerization in the aqueous phase. In the present study the surfactant : monomer molar ratio of 1 : 10

was found to be the optimum condition for both PS and PMMA.

Effects of surface treatments on the wettability of BN surface

The interactions of the treated BN surface with water and epoxy droplets were investigated using the contact angle measurement to assess the changes in the hydrophilicity and wettability of the BN surface. As shown in Table I, the water contact angles

TABLE I
Contact Angles of Untreated- and Treated-BN Particles
Using Water and Epoxy Droplets

Sample	Water contact angle (degree)	Epoxy contact angle (degree)
Untreated BN	86.8 ± 0.30	93.1 ± 0.35
Epoxy	96.5 ± 0.20	–
PMMA-treated	94.0 ± 0.30	70.5 ± 0.30
PS-treated	92.8 ± 0.35	76.4 ± 0.30
GPS-treated	90.0 ± 0.35	79.9 ± 0.30
APS-treated	88.3 ± 0.30	84.9 ± 0.30

TABLE II
Solubility Parameters of Epoxy Resin, Polymethyl
Methacrylate, and Polystyrene

Polymer	Solubility parameter (MPa$^{0.5}$)				
	δ_d	δ_p	δ_h	δ_t	$\Delta\delta$
Epoxy resin	20.4	12.0	11.5	26.31	–
Polymethyl methacrylate	18.6	10.5	7.5	22.63	4.63
Polystyrene	21.3	5.8	4.3	22.49	9.54

δ_t: total solubility parameter, which is defined as: $\delta_t^2 = \delta_d^2 + \delta_p^2 + \delta_h^2$, δ_d dispersive contribution, δ_p polar contribution, δ_h hydrogen bonding contribution.
$\Delta\delta = (\Delta\delta_d^2 + \Delta\delta_p^2 + \Delta\delta_h^2)^{1/2}$.

on untreated BN and epoxy surfaces were 86.8° and 96.5°, respectively. The results show that the untreated BN surface was slightly hydrophilic while the epoxy surface was slightly hydrophobic. The water contact angle of BN surface was found to increase to 92.8° and 94.0° after admicellar-treated with PS and PMMA, respectively, whereas in the cases of treatments with 0.1 wt % APS and 0.1 wt % GPS, the contact angles changed to 88.3° and 90.0°, respectively. The results show that the hydrophilicity/hydrophobicity nature of the admicellar-treated BN surface was closer to that of the epoxy surface than the silane-treated BN surface.

The contact angle of epoxy droplet on untreated BN surface as shown in Table I was found at 93.1° indicating that the epoxy resin could not wet untreated BN surface very well. However, the epoxy contact angle was found to decrease to 76.4° and 70.5° for PS- and PMMA-coated surfaces, respectively. The results show that wettability of the epoxy resin on the BN surface was greatly enhanced after admicellar-coated with PS and PMMA. In the cases of APS- and GPS-treated surfaces, the contact angle was reduced to a lesser extent to 84.9° and 79.9°, respectively.

From the results, it can be concluded that admicellar polymerization can be used to enhance wettabil-

ity of the resin on the filler by coating the filler surface with a thin layer of an appropriate polymer. In the present system of epoxy-BN filler, both PS and PMMA were found to enhance the wettability of the system, and they were more effective than the use of APS and GPS coupling agents. However, when comparing between PS and PMMA, the latter was found to be more effective.

The solubility parameter concept has long been used to predict the compatibility between two materials.[29] The solubility parameter value of a material is derived from its cohesive energy density which depends on the intensity of the interacting forces between molecules in a substance. Hence, theoretically, materials with similar solubility parameter values (δ_t) should have high mutual interactions on the basis of "like dissolves like." Hansen later proposed that the concept had to take into consideration the interactions due to the three different interacting forces, dispersion, polar, and hydrogen-bonding, giving rise to solubility parameter values due to the different forces (δ_d, δ_p, δ_h) known as Hansen solubility parameters (HSP) where $\delta_t = (\delta_d^2 + \delta_p^2 + \delta_h^2)^{1/2}$. To ensure good compatibility, there must be a matching of not only the overall solubility parameter
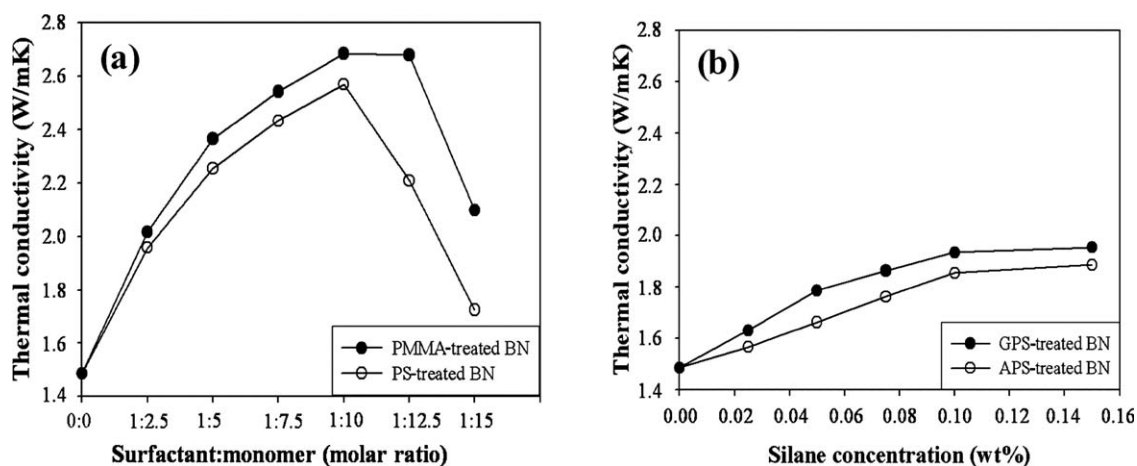

Figure 4 Thermal conductivity of BN-filled epoxy composite treated with varying (a) surfactant : monomer molar ratio, and (b) silane concentration.
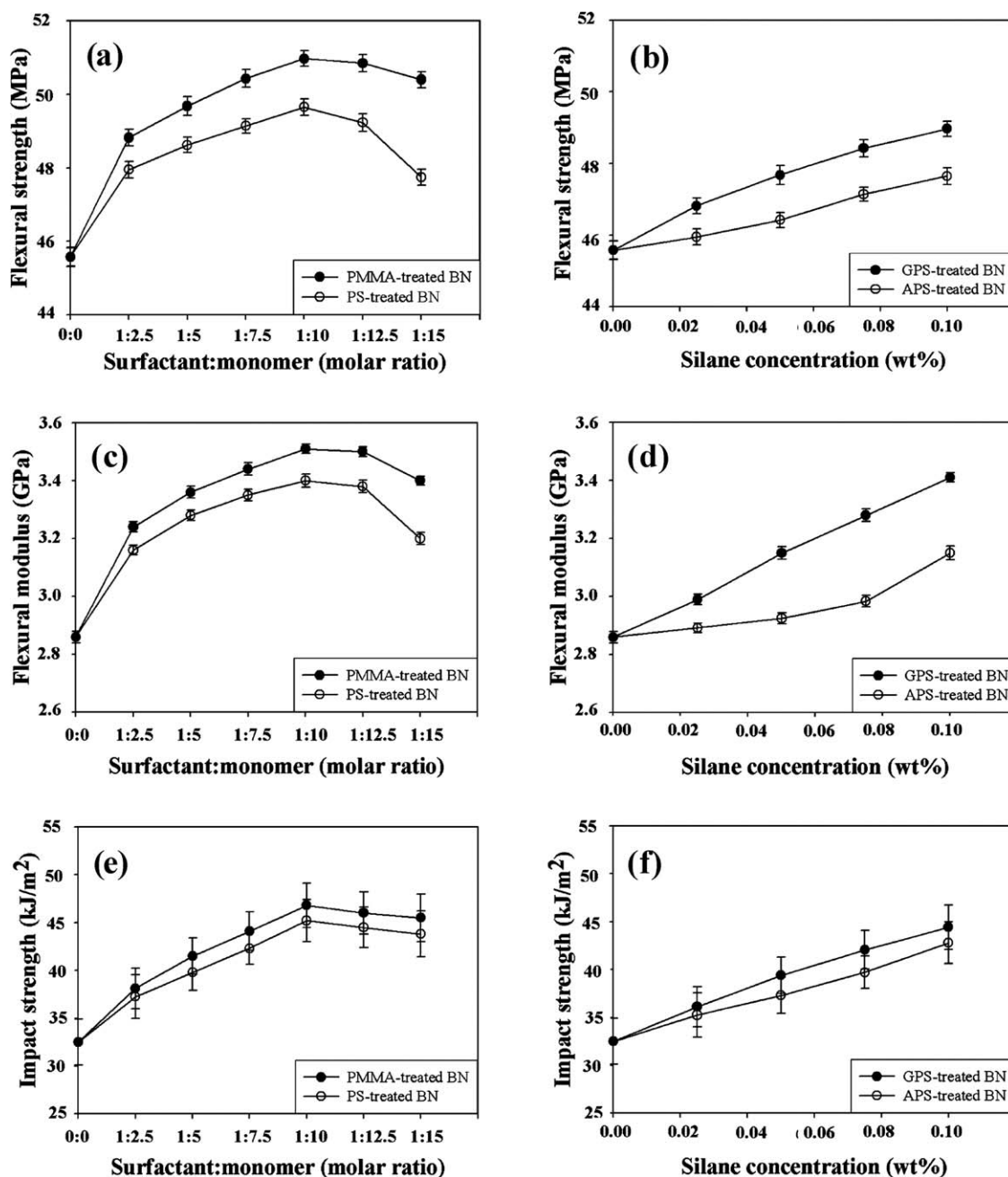
Figure 5 The mechanical properties of surface-treated BN-filled epoxy composite: (a) and (b) flexural strength of admicellar-treated and silane-treated BN-filled epoxy composites, (c) and (d) flexural modulus of admicellar-treated and silane-treated BN-filled epoxy composites, (e) and (f) impact strength of admicellar-treated and silane-treated BN-filled epoxy composites, respectively.

values, but also the solubility parameters derived from the different interacting forces.[30] Table II shows the HSP of epoxy,[30] PS,[31] and PMMA.[32] It can be seen that, although PS and PMMA have similar total solubility parameters of 22, as compared to the value of 26 for epoxy, PMMA is actually closer to epoxy when the three interacting forces were taken into consideration. In this case, the difference in the total solubility parameter value, $\Delta\delta_t$, is defined as the distance joining the two points in

a three dimensional space, or $\Delta\delta_{d,p,h} = (\Delta\delta_d^2 + \Delta\delta_p^2 + \Delta\delta_h^2)^{1/2}$. The value of $\Delta\delta_{d,p,h}$ for PMMA is 4.63 while that of PS is 9.54. Hence, according to the HSP concept, PMMA should be more compatible with epoxy than PS, and this prediction agrees well with the result from contact angle measurements. The results show that HSP can be used to select appropriate polymer to coat on the filler surface by admicellar polymerization to ensure maximum compatibility with the resin in use. It is important,

however, that all the three components of the HSP are taken into consideration.

Effects of BN surface treatments on thermal conductivity of BN-filled epoxy composites

The thermal conductivity of neat epoxy with no filler was found at 0.216 W/mK which is in good agreement with the value quoted in literature.[33] Figure 4 compares the thermal conductivity of 33 vol % BN-filled epoxy composites using admicellar-treated and silane-treated BN particles. The results in Figure 4(a) show that thermal conductivity of both the PS- and PMMA-coated BN-filled epoxy composite increased with increase in the surfactant : monomer molar ratio up to 1 : 10. As the amount of monomer increased, the polymer film formed on BN surface became more uniform and gained in thickness, giving better adhesion between the BN surface and the polymer matrix. Hence, the thermal conductivity of the composite increased with increase in the amount of monomer over this range. The thermal conductivity then started to decline when the amount of monomer was further increased. This is due to the deposition of polymer particles on BN surface as shown in Figure 3(e,f). These particles disrupted the smooth adhesion of the epoxy resin on the admicellar-treated surface leading to lower thermal conductivity. When the effects of admicellar and silane treatments were compared [Fig. 4(a,b)], it was found that admicellar treatment gave much higher thermal conductivity than silane treatment. In the case of admicellar treatment, the thermal conductivity of the composite increased from 1.5 W/mK for the untreated sample to 2.69 and 2.58 W/mK for the PMMA-and PS-treated samples, respectively; an increase of over 70%, while the silane-treated samples gave the maximum value of 1.95 and 1.89 W/mK for GPS- and APS-treated samples, respectively, an increase of only 30%. In the case of silanes, the thermal conductivity of the composite also increased with increase in the amount of silane over the range from 0 to 0.10 wt %, but further increase to 0.15 wt % produced very small further change as can be seen from Figure 4(b). The results indicate that the low effectiveness of silane did not come from the amount of silane used. In comparing between the two silanes, GPS and APS, it was found that GPS was more effective than APS at all concentrations. The results show that the organo group in GPS must be more compatible with epoxy resin than that of the APS. In addition, the greater effectiveness of polymer-coated BN when compared to the silane-coated BN also shows that the two polymers must also be more compatible with the epoxy resin than the two silanes. The results agree well with the contact angle measurements which show that epoxy
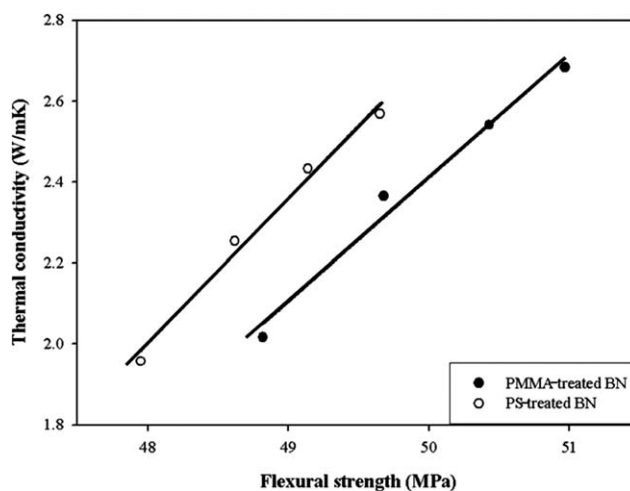
Figure 6 Correlation between thermal conductivity and flexural strength of admicellar-treated BN-filled composite.

resin had better wettability on admicellar-treated BN surface, and the epoxy contact angle on treated-BN decreased in the order: APS, GPS, PS, and PMMA.

Effects of BN surface treatments on mechanical properties of BN-filled epoxy composite

It is well recognized that the mechanical properties of particulate-filled polymer composite depends on the efficiency of stress transfer at the filler–matrix interface which in turns depends on the adhesion strength between the two phases. Improvement in the interfacial adhesion generally leads to an increase in the mechanical properties of the composite. In the present work the adhesion between BN particles and epoxy matrix was enhanced by coating the BN with PS and PMMA by admicellar polymerization. Figure 5(a,c,e) show that the flexural strength, flexural modulus, and impact strength of the admicellar-treated BN-epoxy composite increased with increase in the surfactant : monomer ratio up to the ratio of 1 : 10, after which there was a decline in the properties due to the deposition of polymer particles on BN surface from polymerization of the excess monomer in the aqueous phase. When comparing the mechanical properties of admicellar-treated and silane-treated composites in Figure 5(b,d,f), it was found that, in general, admicellar treatment gave higher mechanical properties than silane treatment in accordance with the greater wettability of epoxy resin on admicellar-treated BN surface.

Since both the thermal conductivity and mechanical properties of a composite depends on the adhesion strength between the filler and polymer matrix, we would expect to see a direct correlation between the two properties. Figure 6 is the plot between the thermal conductivity and flexural strength of the
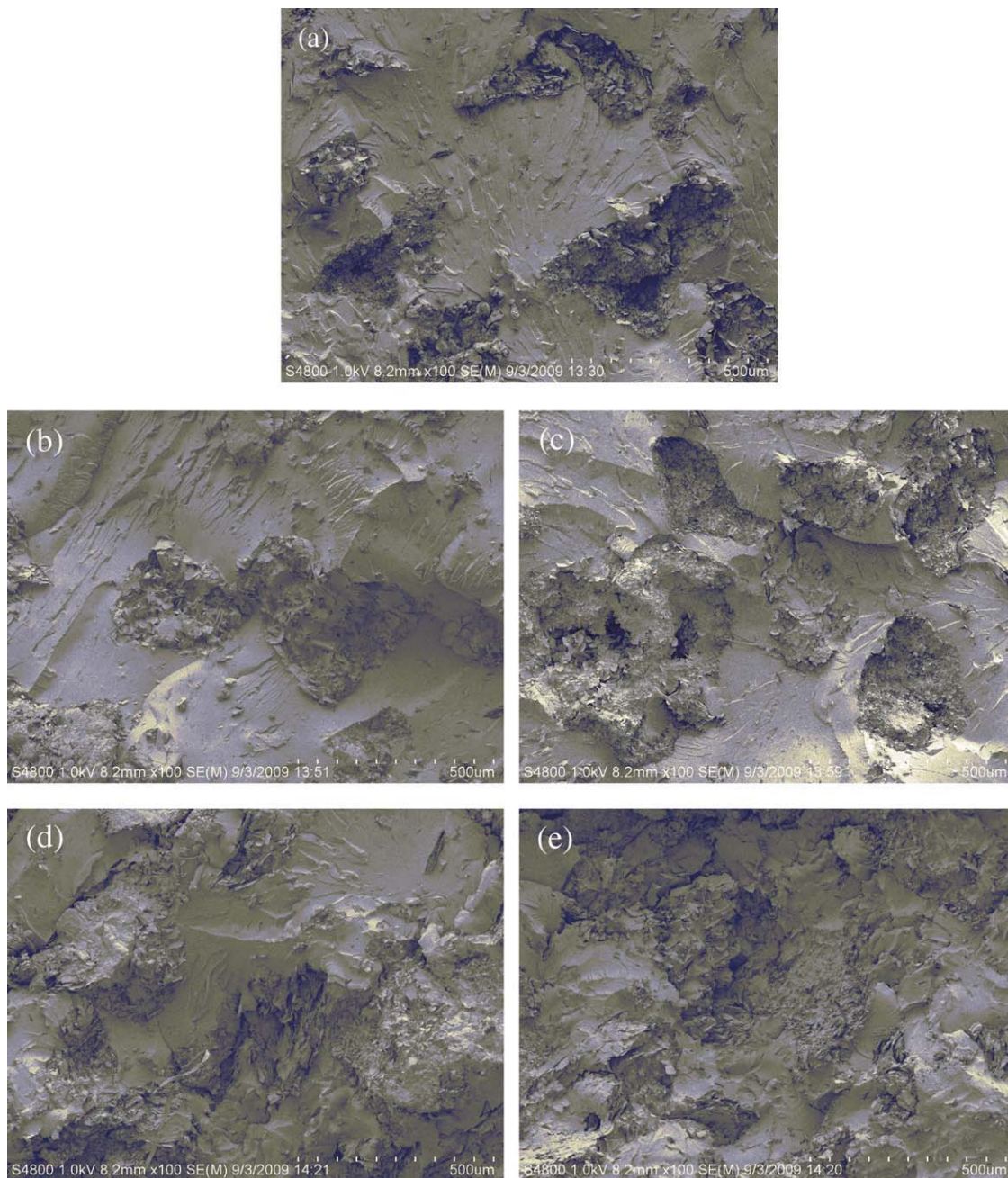
Figure 7 SEM micrographs of the fractured surface of BN-filled epoxy composite using (a) untreated BN, (b) 0.1 wt % APS-treated BN, (c) 0.1 wt % GPS-treated BN, (d) 1 : 10 PS-treated BN, and (e) 1 : 10 PMMA-treated BN. (Magnification ×100). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

composite made from PS- and PMMA-treated BN particles. The results show that there was indeed a direct correlation between the two properties with the PMMA-treated composite giving higher thermal conductivity and better flexural strength. The results confirm that the improvement in the adhesion between polymer-coated BN filler and epoxy matrix plays an important role in both the heat and stress transfers through the interface between the two components.

Morphology of the fractured surface of BN-filled epoxy composite

The effect of surface treatments on the interfacial adhesion between BN and epoxy was studied by examining the fractured surfaces from the flexural tests using SEM. Figure 7(a) shows the fractured surface of composite made from untreated BN which was found to be quite smooth. The poor filler-matrix adhesion resulted in particle pull-out at the interface as evidenced from the presence of both large craters
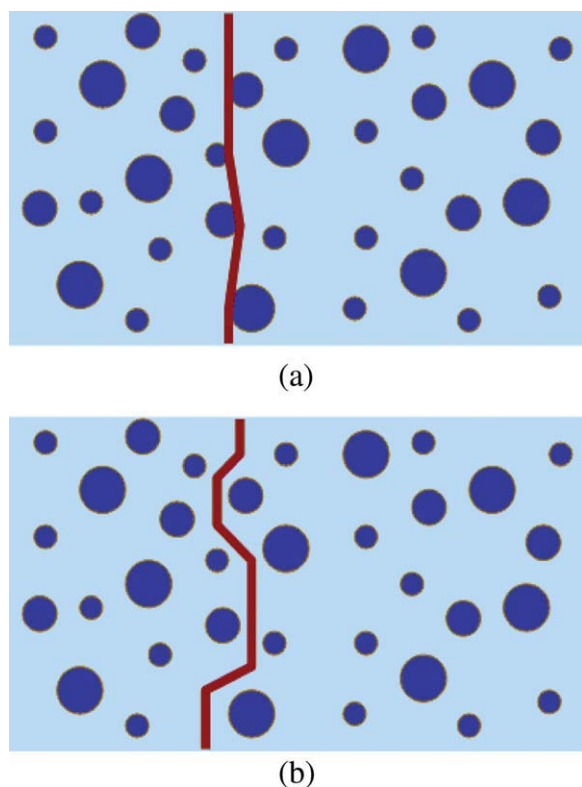
Figure 8 The fracture line in (a) untreated BN-filled composite, and (b) surface-treated BN-filled composite. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

and clean surface of the exposed BN particles at the fractured surface. Figure 7(b,c) show the fractured surfaces of APS- and GPS-treated BN-epoxy composite, respectively. In these cases, the fractured surface remained rather smooth but the BN particles were found to be covered with the epoxy resin indicating better adhesion between the particles and the polymer matrix. On the other hand, the fractured surfaces of composite made from PS- and PMMA-treated BN shown in Figure 7(d,e) were found to be much rougher. There were no craters on the fractured surface and the particle surface was well covered with epoxy resin. According to Spanoudakis and Young, in the case of good bonding between fillers and matrix in the composite, maximum stress will be in the matrix.[34] Hence, in this case, cracks will propagate through the matrix leaving a layer of epoxy resin covering the particles and a rough fractured surface as shown schematically in Figure 8(b). Whereas in the case of poor bonding, fracture occurs from a combination of debonding at the filler–matrix interface and cracking through the matrix resulting in craters and uncovered particles and a relatively smooth fractured surface as shown schematically in Figure 8(a). Hence, the SEM micrographs further confirm that coating of BN particles by admicellar

polymerization of PS and PMMA on the BN surface greatly enhanced the interfacial adhesion between the treated BN particles and the epoxy matrix.

CONCLUSIONS

PS and PMMA were successfully coated on BN particles by admicellar polymerization. The 1 : 10 surfactant : monomer molar ratio was the optimum condition for both polymers. The admicellar treatment was found to increase the interfacial adhesion between the epoxy resin and the treated BN surface as shown by the wettability tests. The thermal conductivity of BN-filled epoxy composite increased from 1.5 W/mK for the untreated BN to 2.69 W/mK for admicellar-treated BN particles. The mechanical properties of the admicellar-treated BN-filled epoxy composite also improved significantly and there was a direct correlation between the improvement in thermal conductivity and the mechanical properties of the composite. The BN surface treatment by admicellar polymerization was found to be more effective in improving the adhesion in BN-filled epoxy composite than the more conventional silane treatment.

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