

CHAPTER V
THE EFFECTS OF BN AGGREGATE SIZE AND ASPECT RATIO ON THE
VISCOSITY OF THE BN-EPOXY MIXTURE AND THE PROPERTIES OF
BN-EPOXY COMPOSITE

5.1 Abstract

Thermal conductivity and mechanical properties of BN-filled epoxy composite as a function of filler content and mixing conditions were studied. Both thermal conductivity and mechanical properties of the composite were found to increase with increases in filler content, mixing speed, mixing time, and mixing temperature. Viscosity of the BN-epoxy mixture was also studied under various mixing conditions. The filler particles stayed in the polymer matrix more as aggregates of various sizes. The mixing action led to a reduction in aggregate size with increase in size dispersity resulting in the improvement in the packing of the particles and their dispersion state. Increases in mixing speed and mixing temperature also led to a better penetration of the polymer matrix into the filler aggregates. A maximum thermal conductivity of 1.68 W/mK was obtained at 37 vol% filler content and the mixing conditions of 300 rpm, 30 min, 30 °C.

Keywords: BN-filled epoxy composite; Thermal conductivity; Flexural property; Impact property; Filler aggregates; Mixing conditions; Viscosity

5.2 Introduction

High thermal conductive composites have become increasingly important in recent years due to the continued miniaturization and increasing power of microelectronic parts. Hence, heat dissipation capability of the electronic packaging becomes critical not only to the life span of device, but also to its performance and reliability [1, 2]. Epoxy resin is an ideal molding compound for encapsulating heat-dissipating electronic components due to its excellent electrical insulation property, ease of processing, and low cost. High thermal conductive fillers are added to the

resin to increase its thermal conductive property. A number of studies have been carried out with varying type of fillers such as metal particles (copper, iron, zinc) and ceramic particles (boron nitride, aluminum nitride) [3, 4]. Metallic fillers have several disadvantages relative to ceramic fillers including high density and susceptibility to oxidation. Boron nitride and aluminum nitride are among the most effective ceramic fillers for these composites due to their high thermal conductivity. However, aluminum nitride is sensitive to moisture while boron nitride does not have this problem [5].

Many studies have been carried out to find ways to maximize the thermal conductivity of particulate-filled composite [6-9]. It is well recognized that important factors that determine the thermal conductivity of a particulate-filled composite consist of the following:

(i) The intrinsic thermal conductivity of the components

A number of ceramic powders such as aluminum nitride, silicon carbide, and boron nitride, are good candidates because of their high intrinsic thermal conductivity.

(ii) The filler content

The filler content is usually determined by the ease of mixing, the optimum properties of the composite, and the maximum packing factor of the filler.

(iii) The packing of the filler

The packing of the filler determines the ease of conductive pathway formation in the composite. It depends on particle size and shape, particle size distribution, and the mixing or processing method.

(iv) The penetration of the polymer matrix into the particulate agglomerates or aggregates.

This depends on the amount of entrapped air and the viscosity of the polymer matrix during mixing.

(v) The interfacial adhesion between the polymer matrix and the filler.

This factor depends on the compatibility between the polymer matrix and the filler, and on any surface treatment applied to the filler.

Several researchers have studied the effects of particle size and shape, and the dispersion state of the filler on the thermal conductivity of the composite. Yung

et al [10] found that the use of multi-modal particle size filler resulted in more efficient packing of the filler as the smaller particles can fill in the spaces between large particles, leading to the formation of greater number of effective conductive pathways and hence, higher conductivity. Bigg [11] found that, at a given volume loading, the greater the aspect ratio of the filler particles, the greater the increase in thermal conductivity. Differing processing methods often lead to different dispersion states of the filler, and hence differences in the number of conductive pathways or networks formed [9]. Bujard [12] compared thermal conductivity of BN-filled epoxy composite prepared by using the conventional mixer and a 3-roll mill. He found that the former method which provided more vigorous mixing gave composite with higher thermal conductivity.

Fillers in powder form normally exist in three types of particles: primary particles, aggregates, and agglomerates [13]. Hence, when these fillers are incorporated in a composite, it is more likely that they will exist in a combination of these three forms rather than in just the form of primary particles. The incorporation of fillers into a polymer matrix generally requires a large amount of mechanical energy which will cause breaking-up of the agglomerates and aggregates. It can be envisaged that changes in the average size of the agglomerates and aggregates from the mixing process can influence the dispersion state of the filler and hence, the properties of the composite. Up till now, although there have been studies on the effects of particles shape, particle size and particle size distribution on the properties of the composite, the studies usually compare the composite properties based on the size and shape of the primary particles, whereas in reality, the particles tend to stay in the form of agglomerates or aggregates. In the present work we study changes in the size and shape of filler aggregates under different mixing conditions and their effects on the thermal conductivity and mechanical properties of a BN-filled epoxy composite.

5.3 Experimental

5.3.1 Materials

The epoxy resin, bisphenol-A-(epichlorohydrin) (EPOTEC YD128), and the curing agent, cycloaliphaticamine (TH7301), were obtained from Aditya Birla Chemicals (Thailand) Ltd. The hexagonal BN (PCTL30MHF) was supplied by Saint-Gobain Ceramics and Plastics, Inc., USA.

5.3.2 Preparation of the Composite

The BN was first evacuated in a vacuum oven at 50 °C for 2 h prior to processing. The desired amount of dried BN was added into 60 mL of epoxy resin contained in a 250 mL plastic beaker. The loading of BN powder was varied from 9%, 16%, 23%, 28%, 33%, to 37% by volume. The mixture was then stirred by a mechanical stirrer (A.L.C. International S.r.l. Class I) at the mixing speed of 80, 150, 250, and 300 rpm with the mixing time of 5, 15, 30, and 60 mins. 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 70 x 90 x 3 mm³ in dimensions. 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 26x26 cm² metal plate and placed in the compression molding machine under 15 tons of loading for curing at 80 °C for 1 h.

5.3.3 Thermal Conductivity Measurement

Thermal conductivity of the composites was measured using a Hot Disk thermal analyzer (Hot Disk AB, Uppsala, Sweden). A minimum of three individual measurements were performed for each specimen (20 x 20 x 3 mm³) with the sensor (3 mm diameter) being 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. The average value of three specimens per sample was reported.

5.3.4 Determination of Particle Size

To determine the particle size in the BN-epoxy mixture, after mixing at the desired conditions, BN particles in epoxy matrix were washed with ethanol to extract BN from the epoxy matrix and the extracted particles were then dried in a

vacuum oven. The dried particles were then placed on a stub and examined using JEOL scanning electron microscopy (SEM) model JSM-5200 (Japan). The narrowest and longest dimensions of BN particles were measured using the Semafore program, and the average aspect ratio was calculated accordingly. A total of 120 particles were examined for each mixing condition and the average value was reported.

5.3.5 The Viscosity Measurement

The viscosity of the BN-epoxy mixture was measured by a Brookfield viscometer (RVDVII) with spindle No. 27 at 25 ± 1 °C according to ASTM 2393-86.

5.3.6 Determination of Flexural and Impact 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. Three specimens ($60 \times 14 \times 3$ mm³) were tested for each sample and the average value was reported. The fractured surface of the specimens from flexural testing was examined by a scanning electron microscope (JSM 5200, JEOL, Japan). Impact strength was determined using a Pendulum Impact Tester (Zwick, Germany) and the testing method was in accordance with ASTM D-256. For each sample, five specimens ($63.5 \times 12.7 \times 3$ mm³) were tested and the average value was reported.

5.4 Results and Discussion

5.4.1 Correlation between thermal conductivity and the filler content of BN-filled epoxy composites

In the present work the filler content was varied from 9, 16, 23, 28, 33, up to 37 vol%. Thermal conductivity of the epoxy resin was found to be 0.22 W/mK which is in good agreement with the value quoted in the literature [8]. At this level of thermal conductivity, the epoxy resin is basically a thermal insulator. However, by incorporating BN particles into the epoxy matrix, the resulting composite exhibited a marked improvement in thermal conductivity as shown in

Figure 5.1. The thermal conductivity was found to increase parabolically with increase in the filler content. The maximum thermal conductivity values of 1.25 and 1.68 W/mK were obtained at 37 vol% and the mixing speeds of 80 and 300 rpm (30 min, 30°C), respectively. These values are much less than the value of 65 W/mK of BN particle indicating that the epoxy matrix plays a major role in the heat transfer process even at the filler content of nearly 40 vol%.

Many models have been proposed in the literatures to predict the thermal conductivity of particulate-filled composites as a function of filler content [14] and a number of researchers have tried to fit their experimental data with the various models with varying degree of success [5, 8, 15-21]. Up till now, it is still not clear which model can best describe the experimental data [22]. In the present work we found that our experimental points shown in Figure 5.2 could fit very well ($r^2 = 0.99$) with such models as the Lewis-Nielsen model, with $A=4$ which corresponds to a nonspherical particle shape, [23] and Kanari model, with $x=3$ which corresponds to an irregular particle shape. [20] These two models take into account the particle shape and the packing factor of particles. Whereas such models as the Maxwell and Bruggeman models [14], which do not take these factors into consideration, did not fit well with our data points. Several researchers also found good fitting with the Lewis-Nielsen [8, 14] and Kanari [19, 21] models. More recently, Pal [22] found that the Lewis-Nielsen could fit reasonably well with a vast number of data available in the literatures. The results therefore support the proposition that the thermal conductivity of a particulate-filled composite depends not only on the filler content but also on such factors as the particle shape and the packing factor of the particles.

5.4.2 Effects of mixing conditions on thermal conductivity of BN-filled epoxy composites

Thermal conductivity of the composites prepared by using different mixing speeds, mixing times, and mixing temperatures was investigated. The results are shown in Figure 5.3. In general thermal conductivity of the composite increased monotonously with increases in mixing speed, time, and temperature. The mixing action had the effect of breaking-up the filler agglomerates via two different mechanisms, the rupture, a large-scale fragmentation, and the erosion, detachment of

small fragments [13]. The results of the breaking-up process can be seen from the SEM micrographs of the BN aggregates after mixing in Figure 5.4 which show a higher amount of smaller particles at the mixing speed 300 rpm when compared to that at 80 rpm. From the results in Table 5.1, it can be seen that there was a reduction in both the average longest and narrowest dimensions of the aggregates with a corresponding increase in the average aspect ratio as the mixing speed, mixing time, and mixing temperature increased. In addition, there was an increase in the spread of the aggregate size as can be seen from the increase in the standard deviation values of the average longest and narrowest dimensions. These changes helped to increase the thermal conductivity as small particles generally give higher thermal conductivity than large particles due to the formation of greater number of conductive pathways [24]. The increase in the amount of smaller particles with greater dispersity also helped in the efficient packing of the particles as the smaller particles can fill in the spaces between larger particles and hence, enhancing the formation of efficient thermal pathways throughout the composite. The results confirm the previous study [10] which shows that the use of multi-sized fillers enhances the formation of thermal conductive network through more efficient packing of the filler leading to higher thermal conductivity. It is interesting to note also that the composite prepared at 80 rpm 60 min 30° C had a thermal conductivity value of 0.97 W/mK, which is much lower than the value of 1.45 W/mK of the composite prepared at 80 rpm 30 min 70° C, despite the fact that the BN particles in both cases had almost the same average dimensions and aspect ratio. This must be due to the better penetration of the epoxy resin at higher mixing temperature apart from the greater dispersity in particle size and aspect ratio which helped in the better packing of the particles. The results further confirm that properties of the composite depend not only on particle size, particle size distribution, and the packing efficiency, but also on the penetration of the resin into the filler aggregates, and the mixing conditions can greatly influence these factors.

The increase in the aspect ratio further enhanced the forming of conductive pathways as filler with higher aspect ratio can form bridges between conductive units, hence the formation of effective network was enhanced leading to higher thermal conductivity [9, 11]. Figure 5.5 compares the aspect ratios of particles

mixed at the speeds of 80 and 300 rpm. It was found that, at the same aspect ratio, composites prepared at higher mixing speed showed much higher thermal conductivity than composites prepared from mixing at lower speed. This shows that thermal conductivity of the composite depends not only on the size, the dispersity, and the aspect ratio of the filler, but also on its dispersion state. The more vigorous stirring at higher mixing speed can result in an overall more homogeneous dispersion of the particles of different sizes. In addition, it can be envisaged that, at high mixing speed, more polymer can be pushed into the spaces inside the aggregates, leading to more efficient heat transfer between the filler and the matrix. From the results, it can be concluded that mixing conditions can influence the particle size, particle size distribution, the aspect ratio of the particle, the dispersion state of the filler, and the penetration of the polymer matrix into the particle aggregates. In general increases in the mixing speed, mixing time and mixing temperature led to a decrease in particle size, increases in particle size distribution and the aspect ratio of the particle, and an increase in the homogeneity of the dispersion state of the filler as well as better penetration of the polymer matrix into the particle aggregates, and these result in a composite with higher thermal conductivity.

5.4.3 Viscosity of the BN-epoxy mixture

Viscosity of a solid suspension in liquid depends not only on the solid concentration but also on the particle size, particle size distribution, and the packing factor of the solid in the liquid. Hence, the viscosity of the BN-epoxy mixture was determined to confirm the effects of the mixing conditions on the properties of the BN particles in the epoxy resin. Figure 5.6 shows the change in viscosity of the mixture with filler content. As expected, the viscosity increased parabolically with increase in filler content. It is interesting to note that the experimental points fit well with Krieger-Dougherty equation which describes the relationship between viscosity of a dispersed suspension and the concentration by volume of the solid [25]. Above the concentration of 33 vol%, there was a sharp rise in both the experimental and predicted viscosity as the filler content approached its maximum packing value when the particles packed so tightly that they could not flow and the viscosity tended to infinity. To confirm the effects of mixing conditions on the particle size, particle size

distribution, and particle dispersion, the viscosity of the mixture under varying mixing condition was examined. It can be seen from Figure 5.7 that viscosity of the mixture increased with increase in mixing speed, mixing time, and mixing temperature. The results were in agreement with the reduction in particle size and the increase in particle size distribution as shown in Table 5.1. The reduction in particle size led to greater filler surface area, hence more friction with the polymer fluid resulting in higher viscosity. The increase in particle size distribution led to better packing of the particles, hence higher viscosity. The results thus confirm that the effects of increasing mixing speed, mixing time, and mixing temperature were to reduce the particle size and increase particle size distribution resulting in better dispersion and packing of the filler.

5.4.4 Effects of the filler content and mixing conditions on the mechanical properties of the composite

5.4.4.1 Flexural properties

When in use, the epoxy composite may be subjected to bending forces, hence, in the present work, the flexural strength and modulus of BN-filled epoxy composite were studied. Figure 5.8 shows that both flexural strength and modulus increased with increase in filler content. The flexural strength increased from 25.7 MPa to 47.6 MPa while the modulus increased from 2.46 to 3.1 GPa as the filler content increased from 0 to 37 vol%. The results show that addition of BN filler into the epoxy resin not only helped in increasing its thermal conductivity but also its flexural properties. Figure 5.9 shows that both the flexural strength and modulus increased with increases in the mixing speed, mixing time, and mixing temperature. The results show that the reduction in particle size, the increase in particle size distribution, and the better dispersion of the filler helped to increase the flexural properties of the composite. The reduction in the particle size led to greater filler surface area in contact with the polymer matrix, resulting in a more efficient stress transfer between the filler and the matrix [26, 27]. The better dispersion state helped in effective sharing of the acting forces leading to higher strength and modulus.

5.4.4.2 Impact strength

From Figure 5.10, the impact strength of BN filled composite was found to increase linearly with filler content from 11.8 KJ/m² in pure epoxy to a maximum value of 36.4 KJ/m² at 33 vol% filler content. It then decreased to 30.1 KJ/m² at the filler content of 37 vol%. The impact strength is an indication of the ability of the composite to withstand a sudden impact. The results show that the filler particles can absorb the impact force more efficiently than the polymer matrix, hence the greater the filler content, the higher the impact strength of the composite. However, at high filler content, the particles tend to form more aggregates. These aggregates may act as stress concentration points, therefore, reduction in impact strength at high filler content is expected [28]. Figure 5.11 shows that, at 28 vol%, the impact strength of BN-filled composite increased with increases in mixing speed, mixing time, and mixing temperature. This resulted from an improvement in particle size distribution and the dispersion state of the BN particles in the epoxy matrix leading to an effective stress sharing among the particles, hence the higher impact strength. In addition, increase in the aspect ratio of the filler also helped to increase the resistance to crack propagation resulting in higher impact strength [26].

5.4.5 SEM micrographs of the fractured surface

To further confirm the effects of the mixing conditions on the properties of the composite, the fractured surface from flexural tests of the samples with 28 vol% filler was examined. Figure 5.12 (a) and (b) show the fractured surface of composite prepared at 80 rpm, 30 min, 30°C, at the magnifications of x100 and x800, respectively. It can be seen that the fractured surface was relatively smooth and the exposed surface of the BN particles was bare. The borderline between the filler particle and the polymer matrix was also well defined. However, as the mixing temperature increased to 70°C (Figure 5.12 (c) and (d)) and the mixing speed increased to 300 rpm (Figure 5.12 (e)-(h)), the fractured surface became rougher, the borderline between the filler particle and the polymer matrix became less defined, and the particle surface at the fractured point was covered more and more with polymer matrix. The rougher fractured surface indicated the better dispersion state of the filler particles, while the less defined borderline and the well-covered particle surface indicated the better penetration of the matrix into the particle aggregates.

The SEM micrographs therefore further confirm the effects of the mixing conditions on the dispersion state and the penetration of the polymer matrix into the particle aggregates.

5.5 Conclusions

In this paper we found that the thermal conductivity of BN-filled epoxy composite increased parabolically with filler content over the concentration range 0-37 vol%. The experimental points were found to fit well with the Lewis-Nielsen and the Kanari models. Increases in the mixing speed, mixing time, and mixing temperature led to a decrease in particle size, an increase in the aspect ratio of the particles, and an increase in the particle size dispersity. These changes led to an increase in the thermal conductivity of the composite due to the increase in the surface area of the filler, the improvement in the packing efficiency and the homogeneity of the dispersion state, and the greater penetration of the polymer matrix into the filler aggregates. The highest thermal conductivity of 1.68 W/mK was obtained from sample mixed at 300 rpm, 30 min, 30 °C, with a filler content of 37 vol%. The viscosity of the filler-polymer matrix mixture was found to increase with increase in filler content, and increases in mixing speed, mixing time, and mixing temperature. The flexural and impact properties of the composite also improved with increases in filler content and mixing conditions.

5.6 References

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Table 5.1 The average dimensions and aspect ratio of BN particles with varying mixing conditions.

Mixing conditions	Avg. narrowest dimension (micron)	Avg. longest dimension (micron)	Avg. aspect ratio	Thermal conductivity (W/mK)
As-received BN	220 ± 61	372 ± 92	1.72 ± 0.44	65
<u>BN mixed at 80 rpm</u>				
5 min	217 ± 70	368 ± 103	1.74 ± 0.64	0.77
15 min	216 ± 72	365 ± 106	1.75 ± 0.52	0.82
30 min	215 ± 75	361 ± 110	1.77 ± 0.39	0.86
60 min	198 ± 80	353 ± 142	1.85 ± 0.51	0.97
<u>BN mixed at 300 rpm</u>				
5 min	215 ± 72	366 ± 120	1.76 ± 0.48	0.92
15 min	211 ± 78	358 ± 139	1.78 ± 0.35	1.09
30 min	193 ± 88	346 ± 152	1.82 ± 0.31	1.25
60 min	190 ± 90	338 ± 158	1.86 ± 0.41	1.30
<u>BN mixed at 80 rpm</u>				
<u>30 min</u>				
30 °C	215 ± 75	361 ± 110	1.77 ± 0.39	0.86
50 °C	211 ± 80	357 ± 120	1.76 ± 0.55	1.16
70 °C	197 ± 85	352 ± 148	1.85 ± 0.74	1.45
<u>BN mixed at 300 rpm</u>				
<u>30 min</u>				
30 °C	193 ± 88	346 ± 152	1.82 ± 0.31	1.25
50 °C	191 ± 90	343 ± 159	1.84 ± 0.64	1.43
70 °C	188 ± 98	335 ± 163	1.87 ± 0.82	1.57

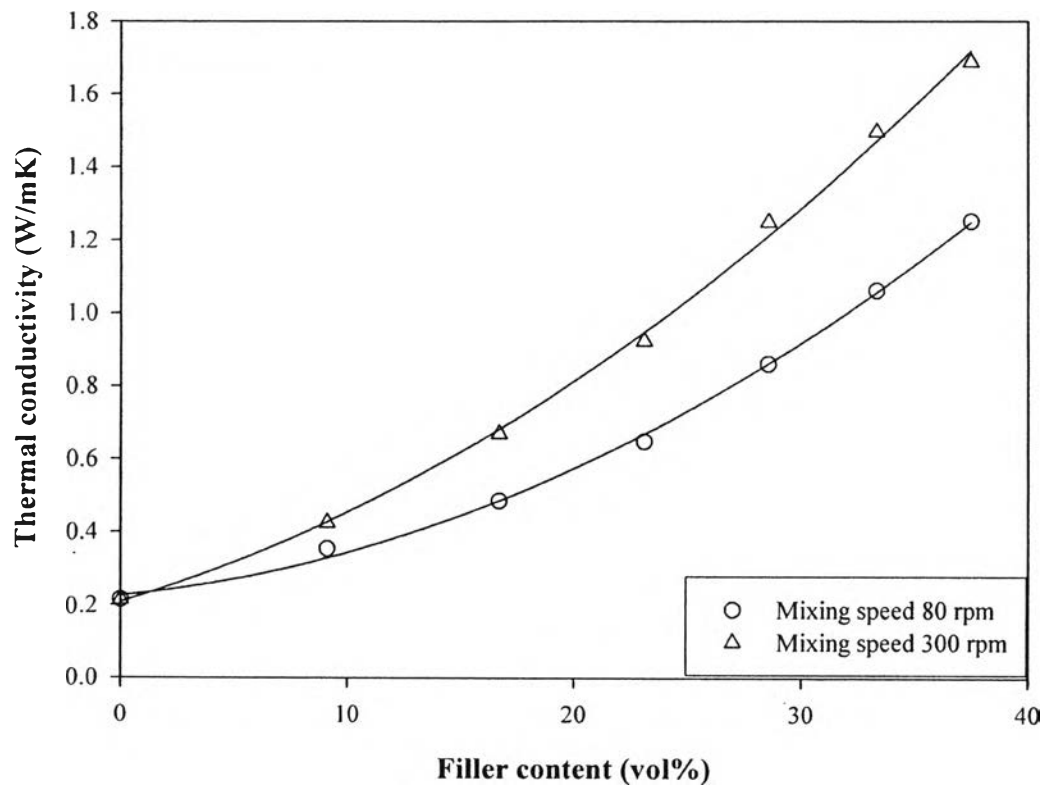
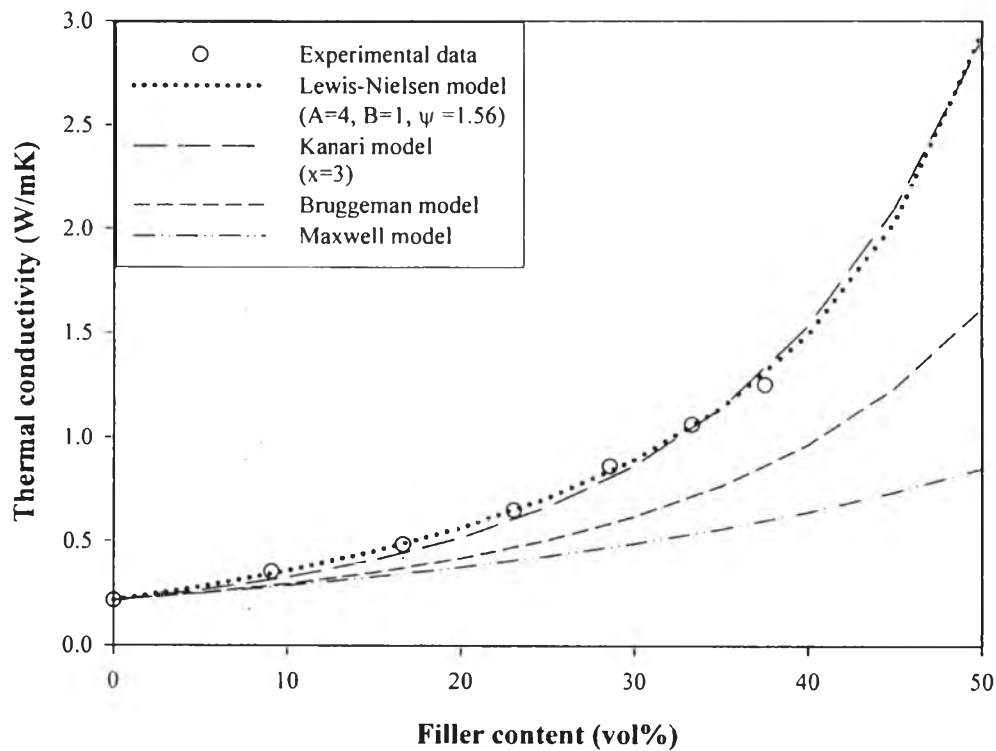


Figure 5.1 Thermal conductivity of BN-filled epoxy composites at different filler contents (mixing time 30 min, mixing temperature 30 °C).



Maxwell model

$$k = k_p \frac{[2k_p + k_f + 2V_f(k_f - k_p)]}{2k_p + k_f - \phi(k_f - k_p)}$$

Bruggeman model

$$1 - \phi = \frac{(k_f - k)(k_p/k)^{1/3}}{k_f - k_p}$$

Kanari model

$$1 - \phi = \frac{(k - k_f)(k_f/k)^{1/(1+x)}}{k_p - k_f}$$

Lewis-Nielsen model

$$\frac{k}{k_p} = \frac{(1 + AB\phi)}{(1 - B\phi\psi)}, \quad B = \frac{(k_f/k_p - 1)(k_f/k_p + A)}{k_f/k_p + A}$$

where

k is Thermal conductivity of composite.

k_p is Thermal conductivity of polymer matrix.

k_f is Thermal conductivity of filler.

ϕ is The volume fraction of filler.

x is The constant determined by sphericity of the filler.

A is Factor depending on particle geometry.

ψ is The maximum packing density of filler.

Figure 5.2 The fitting of experimental data with Lewis-Nielsen, Kanari, Bruggeman, and Maxwell models (mixing speed 80 rpm, mixing time 30 min, mixing temperature 30 °C).

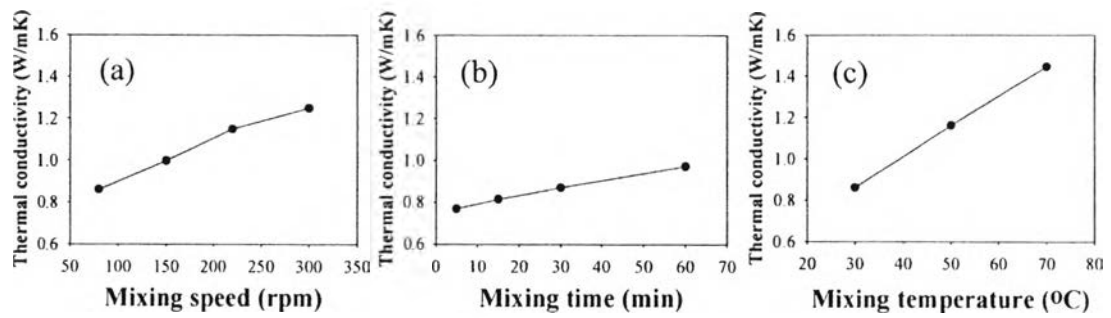


Figure 5.3 Thermal conductivity of 28 vol% BN-filled epoxy composites at varying (a) mixing speed (30 min, 30°C), (b) mixing time (80 rpm, 30°C), and (c) mixing temperature (80 rpm, 30 min).

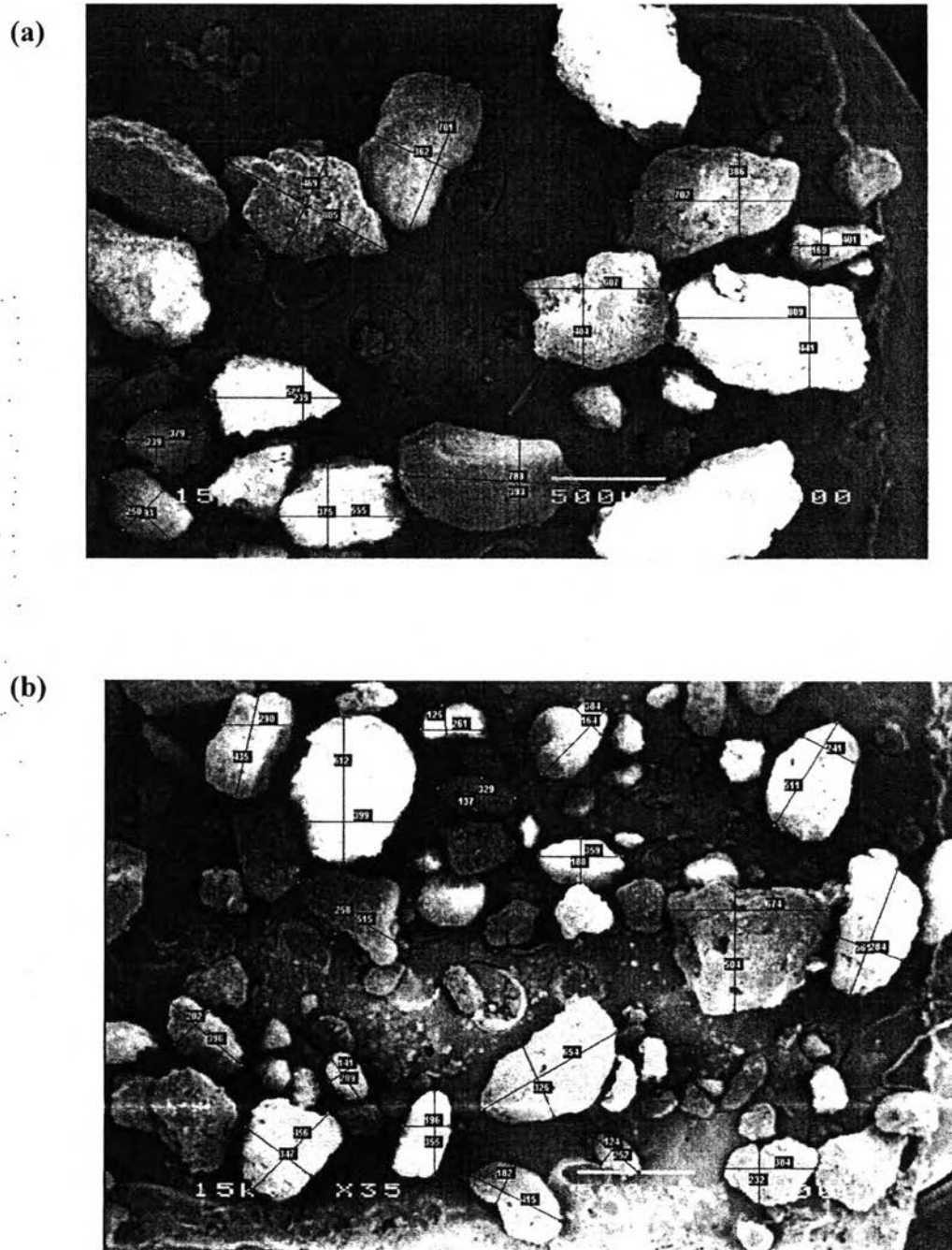


Figure 5.4 SEM micrographs (x35) of BN particles in 28 vol% BN-filled composite at the mixing speed of (a) 80 rpm and (b) 300 rpm (30 min, 30 °C).

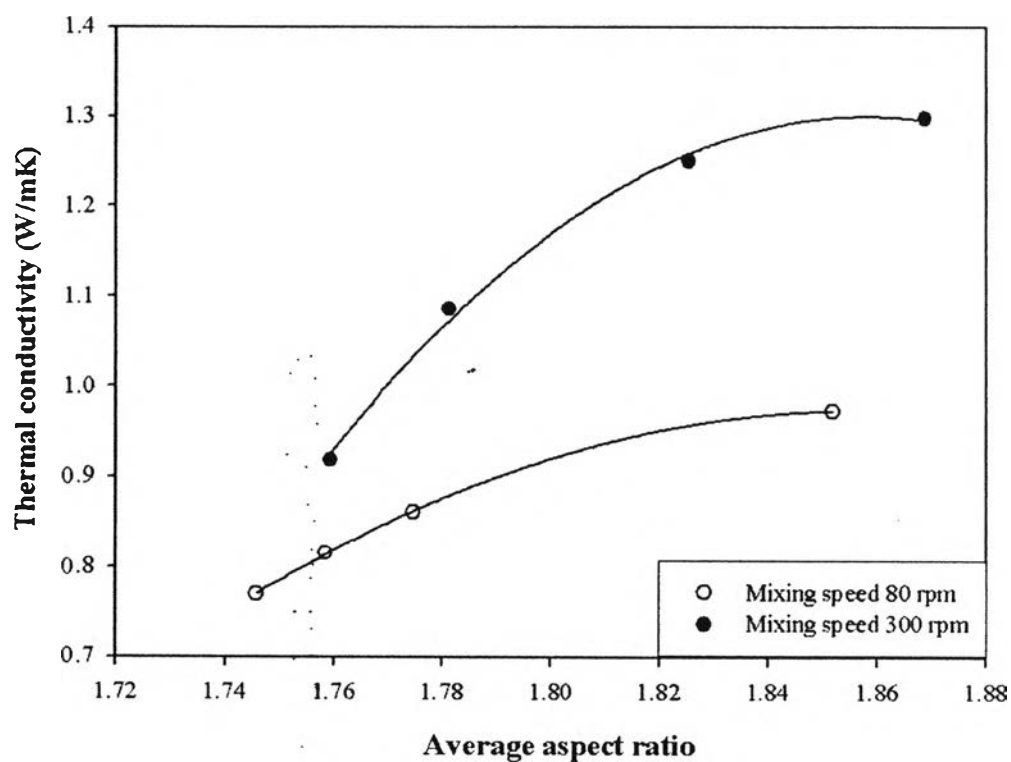


Figure 5.5 Correlation between the thermal conductivity and the average aspect ratio of extracted BN particles.

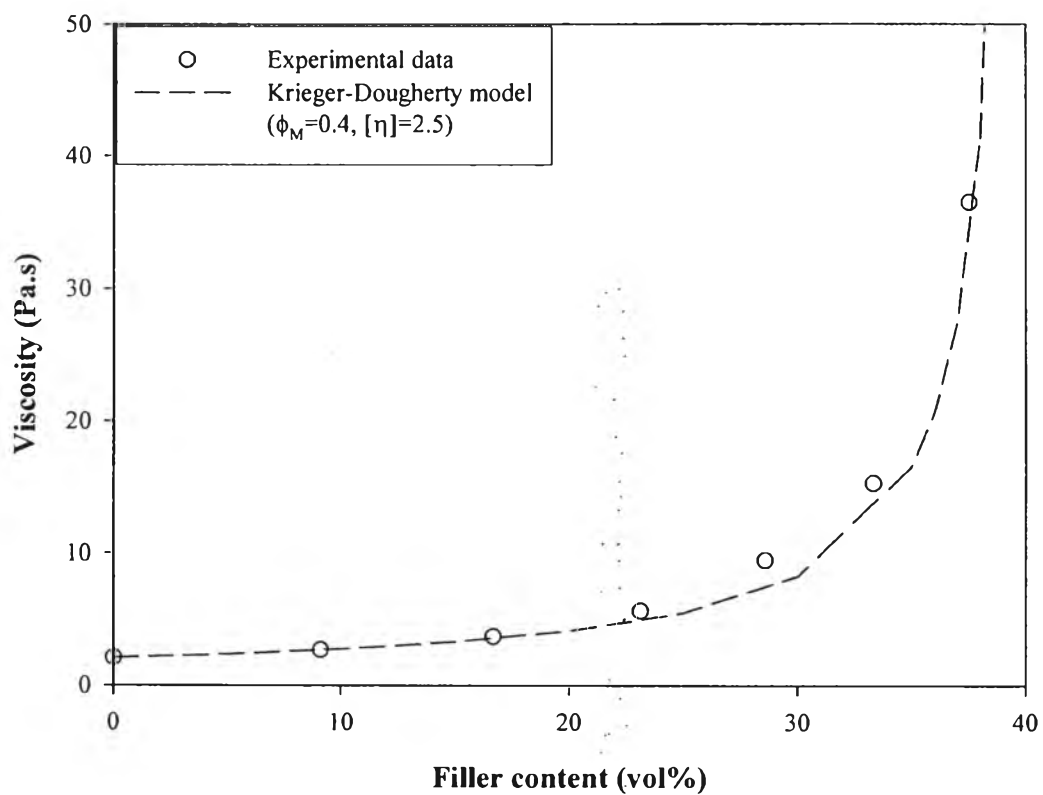


Figure 5.6 Viscosity of BN-epoxy mixture at different filler contents and the fitting of the experimental data with Krieger-Dougherty model.

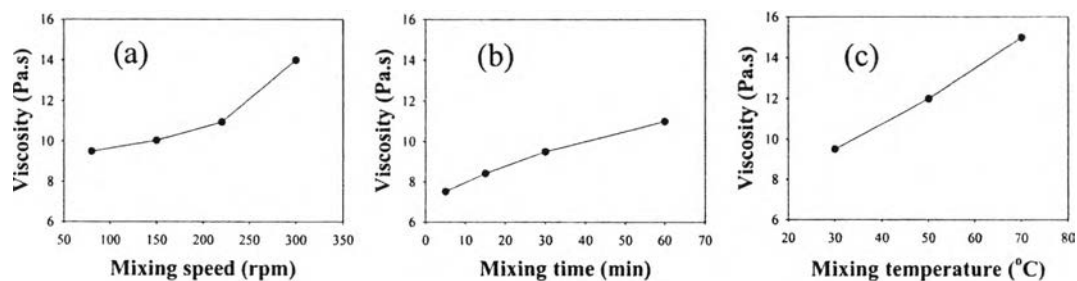


Figure 5.7 Viscosity of 28 vol% BN-filled epoxy suspension with varying (a) mixing speed (30 min, 30°C), (b) mixing time (80 rpm, 30°C), and (c) mixing temperature (80 rpm, 30 min).

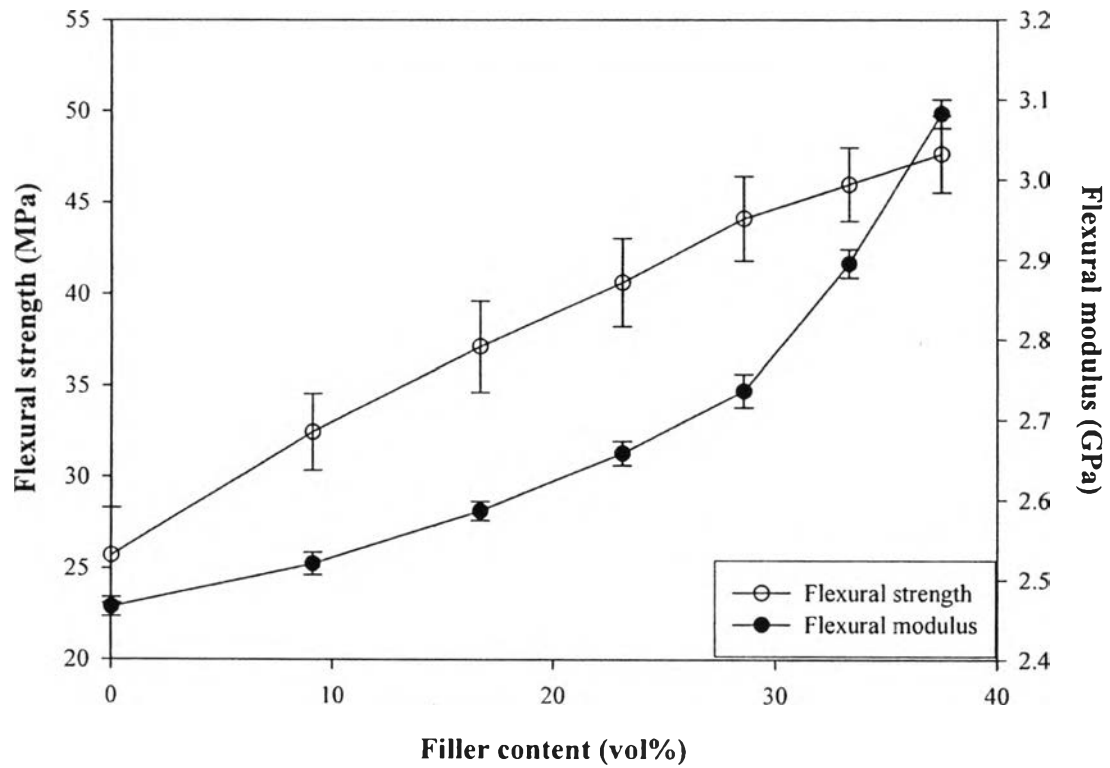


Figure 5.8 Flexural properties of BN-filled epoxy composite at different filler contents

(mixing speed 80 rpm, mixing time 30 min, mixing temperature 30 °C).

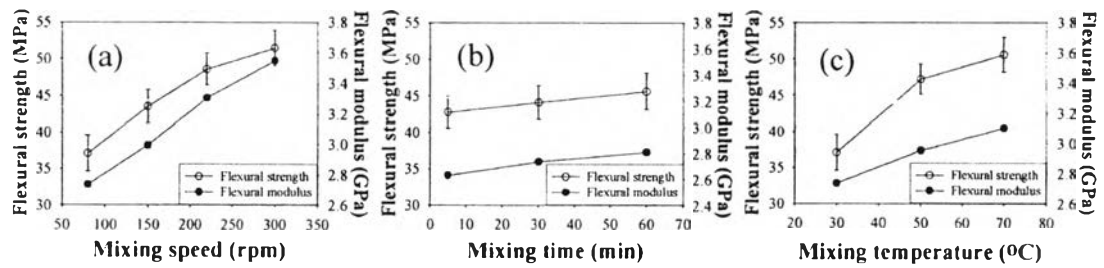


Figure 5.9 Flexural properties of 28 vol% BN-filled epoxy with varying (a) mixing speed (30 min, 30°C), (b) mixing time (80 rpm, 30°C), and (c) mixing temperature (80 rpm, 30 min).

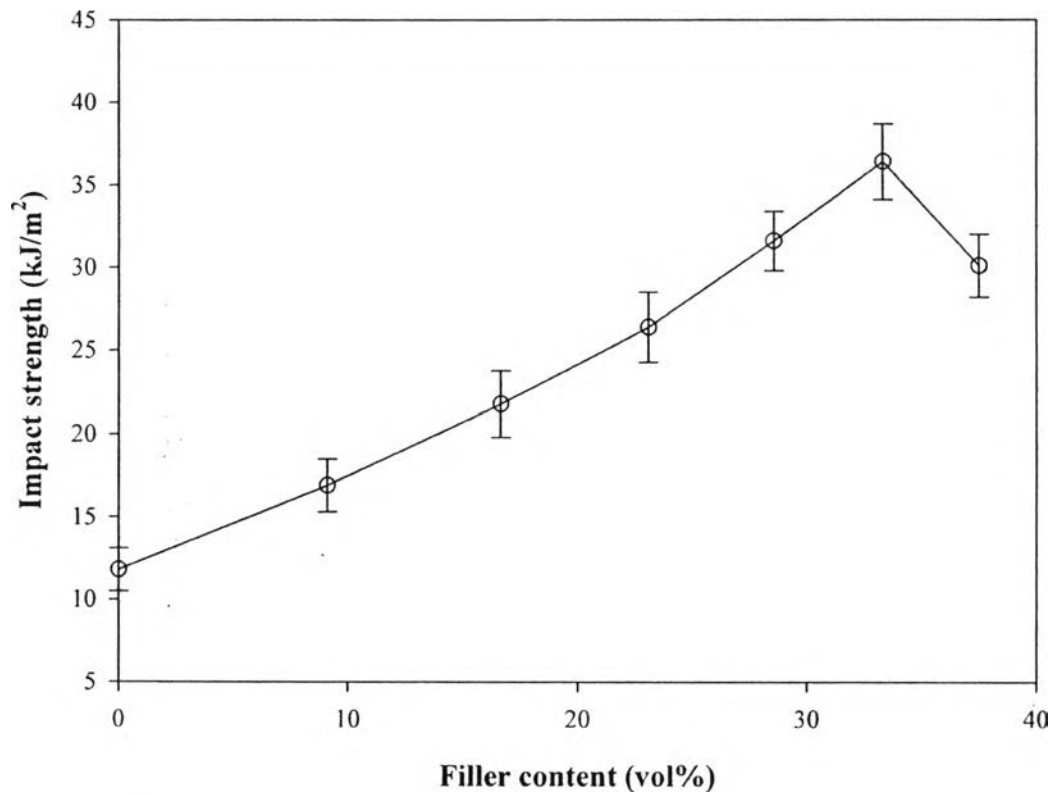


Figure 5.10 Impact strength of 28 vol% BN-filled epoxy composite at different filler contents (mixing speed 80 rpm, mixing time 30 min, mixing temperature 30 °C).

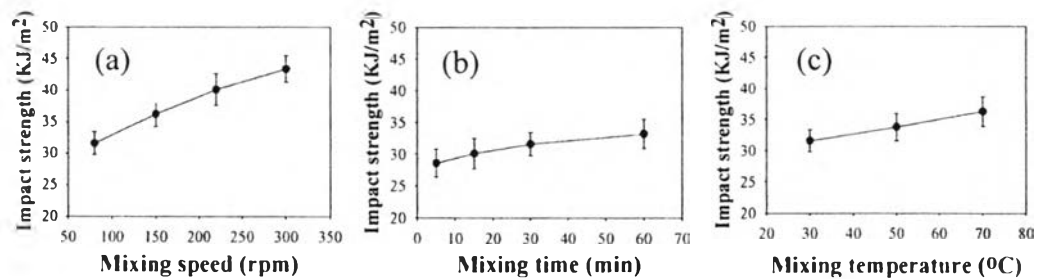


Figure 5.11 Impact strength of 28 vol% BN-filled epoxy with varying (a) mixing speed (30 min, 30°C), (b) mixing time (80 rpm, 30°C), and (c) mixing temperature (80 rpm, 30 min).

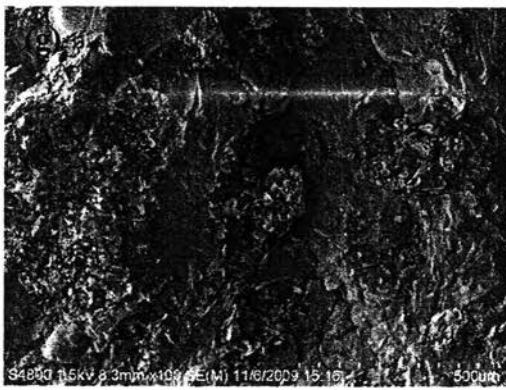
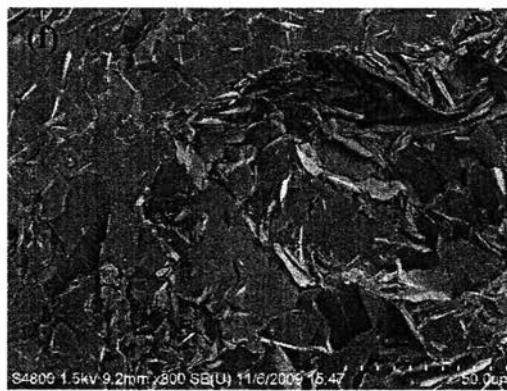
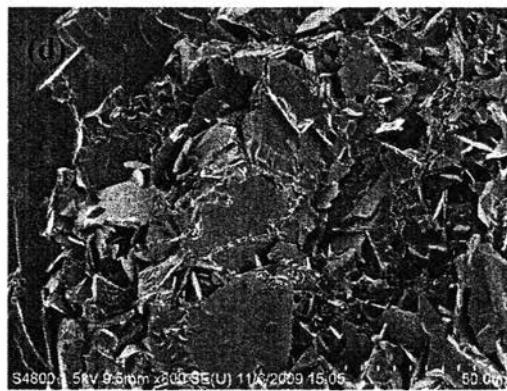
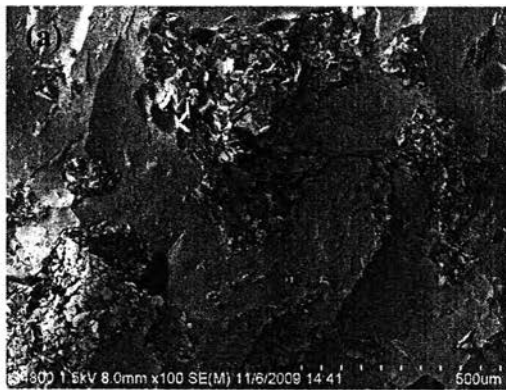


Figure 5.12 SEM micrographs of the fractured surface of 28 vol% BN-filled epoxy composite prepared by mixing at (a) and (b) 80 rpm, 30 min, 30°C at x100 and x800, (c) and (d) 80 rpm, 30 min, 70°C at x100 and x800, (e) and (f) 300 rpm, 30 min, 30°C at x 100 and x800, (g) and (h) 300 rpm, 30 min, 70°C at x 100 and x800.

