Effect of SiC Whisker on Benzoxazine-Epoxy-Phenolic Ternary Systems: Microwave Curing and Thermomechanical Characteristics

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ABSTRACT: Microwave radiation at 2.45 GHz with variable power input was investigated as a tool to facilitate the curing reaction of benzoxazine-epoxy-phenolic molding compound i.e., BEP893. Dielectric filler for microwave coupling was silicon carbide whisker (SiC_w). Factors such as whisker loading and input irradiation power were found to have a profound effect on the microwave heating of the BEP893 particularly on the rate of temperature rise and maximum heating temperature. The SiC_w loading of 10% by weight with the microwave irradiation condition of 300 W for 10 min renders the ultimate curing of the molding compound. Significant reduction in processing time of the microwave cured sample compared with the conventional heat cured sample i.e., 150 min at 200°C using conventional heating is the key benefit of this technique. Mechanical properties of the microwave cured and conventional heat cured samples show similar characteristics with slightly lower T_g in the microwave cured samples. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 105: 1968–1977, 2007

Key words: composites; mechanical properties; thermal properties; crosslinking differential scanning calorimetry (DSC)

INTRODUCTION

Benzoxazine resin (BA) was developed as a high flow, low void resin system with a capability of forming thick samples of either filled or unfilled systems. The resin can be synthesized via a simple and cost-effective solventless technology.[1] In addition, molecular design flexibility of the resin comparable to that of epoxy or polyimide renders wide range of properties of the polymer that can be tailor-made.[2–4] The resin has been shown to possess some useful properties such as ease of processing due to its self-polymerizability upon heating via ring-opening polymerization thus giving no volatile by-products. The polymer shows near-zero shrinkage upon polymerization as well as possesses relatively high T_g and good thermal stability.[5–8] Ishida and Rimdusit have reported the use of BA-m type polybenzoxazine as a matrix for boron nitride filler to obtain a highly filled composite system with high value of thermal conductivity.[9,10] Recently, highly filled wood composites from BA-a type polybenzoxazine with relatively high modulus comparable to natural wood has also been reported.[11]

Those high performance composite properties are attributed to the ability of the low viscosity BA resin to accommodate very high filler loading i.e., up to 75% by volume as well as its good adhesive properties. Moreover, the compatibility of the polybenzoxazine with various resins renders a large number of polymer alloys or copolymers covering wide range of properties.[12–21] The hybrid systems based on BA, epoxy, and phenolic resins are of particular interest in this investigation since the systems show synergistic behaviors in some of their properties in addition to their excellent processbility and high reliability of the cured samples.[17,18] In this investigation, the well-characterized ternary systems namely BEP893 which is the resin mixture of BA resin (B), epoxy resin (E), and phenolic resin (P) at the mass ratio of 8:9:3 is used as a matrix. The processability as well as the cured properties of the resin has already been reported in our previous work.[17]

Microwave energy has been an attractive heating source for material processing due to its capability to interact directly with molecules i.e., by raising their rotational energy level and thus the temperature. The consequence is a more uniform and faster heating of the materials than traditional ways of heat

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conduction or convection.[22–25] The technique has already been utilized in various systems such as to accelerate reaction kinetics in the drug development industry, to cure plywood cement, or to vulcanize rubber in the tire industry.[22] There are a number of investigations on the use of microwave to cure thermosetting resins particularly in epoxy systems and its molding compounds. Some reports e.g., on the epoxy systems, showed promising results in the enhancement of the processing time using microwave radiation comparing with the traditional thermal curing.[26,27]

In this investigation, we utilize microwave energy for the curing process of BEP893 resin filled with inductive or dielectric filler i.e., silicon carbide whisker (SiC_w). The effect of filler loading and input radiation power on microwave coupling of this molding compound as well as the composite properties comparing with those obtained from the conventional heat cure method is to be investigated.

EXPERIMENTAL

Materials

The 4,4′-isopropylidenediphenol and aniline were purchased from Kanto Chemical Co., Inc. (Tokyo, Japan). Paraformaldehyde was from Merck Chemical Ltd. (Notthingham, UK). BA resin based on the above reactants was synthesized using a solventless method as reported elsewhere.[1] The as-synthesized monomer is a clear yellowish solid at room temperature and can be molten to yield a low viscosity resin at about 80°C. The monomer was ground to fine powder and was kept in a refrigerator prior to use. Bisphenol F type epoxy resin (YDF-170) from Tohto Chemical Co., Ltd. (Tokyo, Japan) is a clear liquid at room temperature and was used as-received. Phenolic novolac (PR1501) from Hitachi Chemical Co., Ltd. (Tokyo, Japan) was utilized as an initiator of BA resin's ring opening reaction.[17] In this investigation, the ternary mixture with the mass ratio of BA resin: epoxy resin: phenolic resin of 8:9:3 i.e., BEP893 was chosen as a matrix for SiC_w due to its well characterized properties to yield a relatively high thermal stability matrix.[17]

SiC_w (TWS-200) from Tokai Carbon Co., Ltd. (Tokyo, Japan), having average diameter of 0.5 μm and average length of 30 μm, was used as a filler for this investigation. The filler has a reported density of 3.20 g cm^{-3} and a dielectric constant of about 40.[28,29]

Sample preparation

BEP893 resin was prepared by melt-mixing the three monomers at 80°C for 20 min. The resulting homogeneous and low viscosity mixture can be compounded with SiC_w at this stage. The BEP893 is solid at room temperature and can be ground and kept in a refrigerator for future use.

Molding compound of SiC_w and BEP893 was prepared by dry mixing of the desired amount of the resin and the whisker. The powder mixture was then heated at 80°C and mechanically blended to yield a uniform suspension of the molding compound. About 5 g of the molding compound was used for microwave heating or conventional heating by compression molding. The fully cured composites by compression molding were obtained using temperature of 200°C for 180 min under the hydraulic pressure of 0.1 MPa.

The microwave heating experiment was carried out using a JRC industrial microwave machine model NJA2103A. The operating frequency is 2.45 GHz with variable input power from 0 to 1.2 kW. A microwave generator (synthesizer and amplifier) supplies the radiation energy to a horn antenna which is directed to the surface of the sample. The reflected microwave energy can be measured by a power monitor and was minimized by a turner fitted on a waveguide before a horn antenna. The nonreflected part of the microwave radiation is absorbed by the sample resulting in heating of the material. The evolution of surface temperature of the sample was monitored using infrared thermometer attached on the sidewall of the microwave apparatus. The temperature of the environment inside the microwave applicator was also recorded using an AMOTH8000 fiberoptic thermometer from Anritsu Meter.

Sample characterization

Dielectric property measurement

A LCR meter from Yokogawa-Hewlett–Packard model HP 4284A (20 Hz–1 MHz) equipped with dielectric test adaptor model 16451A (Yokogawa-Hewlett–Packard), having parallel-plate micrometer-typed electrodes was used to determine the capacitance, a corresponding dielectric constant, and a loss tangent of dielectric material. The measurement was performed at room temperature. The test material was in the form of disk shape with a diameter of about 35 mm and a thickness of about 1 mm. A thin layer of high vacuum silicone grease was applied on both specimen surfaces to ensure good contact and to eliminate air gap between the sample and the electrodes.

Differential scanning calorimetry

A differential scanning calorimeter model DSC3100 from MAC Science was utilized to investigate curing behaviors as well as to determine the transition temperature of both filled and unfilled BEP893. The

mass of the sample is ~10 mg. The sample was put in an aluminum pan with lid and was scanned using the heating rate of 10°C min^{-1} from room temperature to 320°C under N_2 purging.

Thermomechanical analysis

Linear thermal expansion coefficient (LCTE) of a specimen was determined using a Seiko Instruments thermomechanical analyzer (model TMA/SS120C) with sample size of 10 × 5 × 1 mm^3. The measurement was performed under tension at 10 g (force control mode). The temperature was scanned twice from 30 to 200°C at a heating rate of 2°C min^{-1}. The value of the LCTE was recorded on the second run and was averaged between 40 and 80°C.

Dynamic mechanical analysis

Dynamic mechanical thermograms of the polymer and its composites were obtained using a dynamic viscoelastic analyzer model DVA-200 from IT Keisoku-Seigyo. The test was performed under tension mode using 1 kgf load cell. The strain amplitude was 0.2% and the frequency used was 10 Hz. The sample was heated at the rate of 2°C min^{-1} from room temperature to 280°C. The samples were in the dimension of 25 × 5 × 1 mm^3.

Scanning electron microscope

The fracture surface morphology of both microwave cured and conventional heat cured samples was obtained by scanning electron microscopy (Akashi Beam Technology model Alpha-30A) at the magnification of 1000× or 2000× with an acceleration voltage of 20–25 kV. Samples were coated with a thin film of gold using a JEOL ion sputtering device (model JFC-1100E) for 4 min to obtain a thickness of ~300 Å before micrographs of the magnified fracture surfaces of the composite were taken.

RESULTS AND DISCUSSION

Characteristics of BEP893 resin and microwave heating

Figure 1 illustrates the DSC thermograms of the uncured and fully cured neat BEP893 matrix. The figure clearly exhibits the thermally curable behavior of this BEP893 resin. The resin's curing exotherm starts at about 140°C with the peak maximum at 243°C. The curing enthalpy of the resin determined from the area under the exothermic peak is 210 J g^{-1}. The fully cured BEP893 exhibits a glass transition temperature (T_g) of about 145°C. This information is essential for microwave heating since the target heating temperature due to microwave irradiation should be well above 140°C to be able to cure this BEP893 resin. In practice, the curing temperature in the range of 180–200°C is preferable to achieve fast curing with minimum thermal degradation of this polymer.[17]

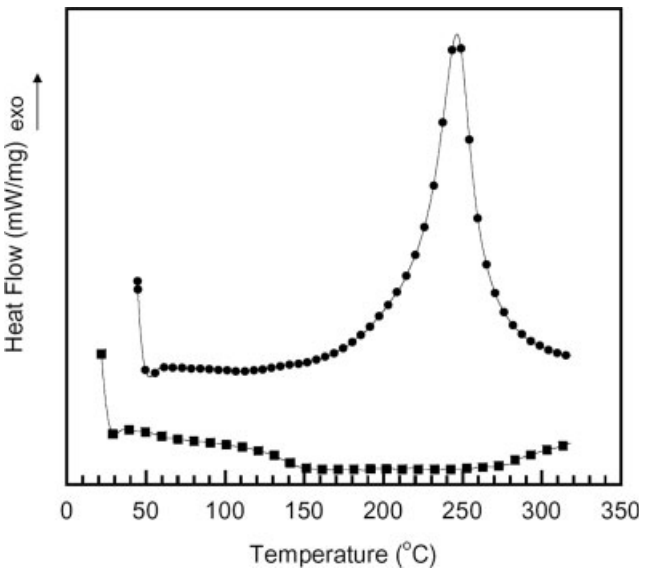

Figure 1 DSC thermograms of the uncured and fully cured BEP893: (●) uncured BEP893, (■) fully cured BEP893.

Figure 2(a,b) exhibit the temperature rise curves due to microwave irradiation of the three starting resins namely BA, epoxy, and phenolic resins, as well as their ternary mixture under this investigation i.e., BEP893 resin. The temperature rise curves as a function of time of the three starting monomers are shown in Figure 2(a). The microwave power used in this investigation was fixed at 1 kW. From the figure, it is evident that only epoxy resin could substantially couple with microwave; thus, rendering the rapid temperature rising beyond 180°C within few minutes. Whereas both BA and phenolic novolac resins show a much slower rate of temperature rising, i.e., the slope of the plot, with the obtained maximum temperature of ~130°C. As a consequence, BA resin solely cannot be cured by microwave irradiation because its maximum attainable temperature, even at a relatively high microwave input of 1 kW, is still rather low for the initiation of its ring-opening polymerization reaction.

The outstanding microwave coupling capability of the epoxy resin is attributed to its relatively polar nature of the resin and makes it one of the most investigated resins for processing by microwave.[24–27] Therefore, the incorporation of substantial amount of epoxy resin into the BA resin should enhance the microwave coupling capability of the resulting mixture. However, in reality, the maximum fraction of

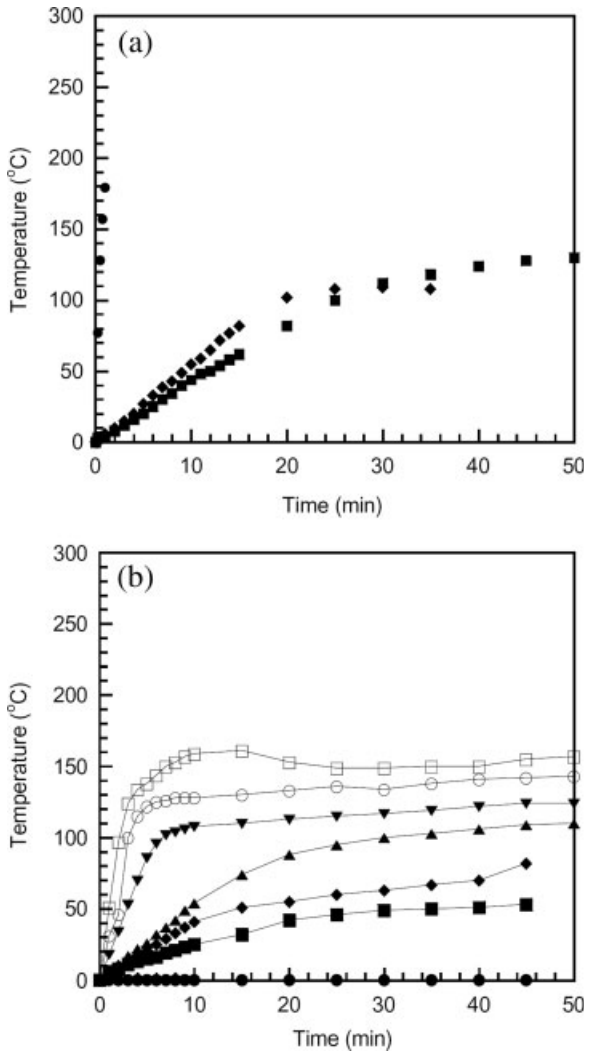

Figure 2 (a) Temperature rise curves at 1 kW of three starting monomers: (●) Bisphenol F epoxy resin, (■) Phenolics resin, and (◆) BA-a benzoxazine resin. (b) Temperature rise curves upon microwave irradiation of BEP893 resin at various microwave input powers: (●) 100 W, (■) 200 W, (◆) 300 W, (▲) 400 W, (▼) 500 W, (○) 600 W, and (□) 1 kW.

the epoxy required is normally limited by the resulting properties of the cured sample. In this work, BEP893 is evaluated for microwave processing due to its relatively good overall cured properties as reported in our previous work.[17]

The temperature rise curves of BEP893 resin at varied microwave input power ranging from 100 W to 1 kW are plotted in Figure 2(b). In this figure, we can see that this BEP893 resin clearly shows improved microwave coupling ability compared with that of the neat BA resin. It was also observed that the initial rate of temperature rise of the resin, which is related to the initial slope of each curve, as well as the maximum achievable temperature rise are increased with increased microwave input power. However, the resin's temperature rise even at the evaluated input power up to 1 kW was found to be only about 160°C, which is still rather low to achieve at fast curing of the BEP893 resin.

Effect of SiC_w on microwave processing of BEP893

Though providing good overall cured properties, the limited microwave coupling ability of the BEP893 resin led to the use of appropriate dielectric fillers to further enhance its microwave heating property. Recently, various types of dielectric fillers have been incorporated in polymeric matrices to further assist in its microwave processing or heating including SiC_w in polypropylene,[30] in EPDM,[31] and in polybenzoxazine[32]; or aluminum powder in epoxy,[33] carbon black in epoxy[34]; as well as polyaniline in polyethylene.[35] Kitano et al.[36] reported the use of electro-conductive and inductive fillers such as short carbon fiber and SiC_w for the heating or melting process of polyethylene. They suggested that the benefit of using SiC_w as microwave-assisted coupling filler was due to its effective heating as well as less sparking event during microwave irradiation comparing with the use of short carbon fiber or carbon black. The other potential benefits of using SiC_w as filler are its excellent reinforcing behavior[30,31] and its reported outstanding toughening ability, particularly in the rigid matrix such as in many ceramics composite systems.[37–40] The filler is, therefore, chosen as the dielectric filler for our BEP893 resin.

Figure 3(a–c) depict the effect of SiC_w contents, i.e., 5, 10, and 15% by weight, on the temperature rise curves of BEP893 at various input microwave irradiation power. From these figures, we can clearly see that the presence of the SiC_w has a dramatic effect on the heating of the BEP893 molding compounds. In Figure 3(a), two significant features were observed from using this inductive filler. First, to reach the target temperature for fast curing of BEP893, i.e., 180–200°C in this case, relatively low SiC_w content of only 5% by weight was found to be sufficient. Moreover, the input power needed to heat the sample above the target temperature was substantially reduced from 1 kW to only about 400 W. From Figure 3(b), the SiC_w loading of 10% by weight with the irradiation power of 100–300 W was found to be sufficient for the microwave processing of the BEP893 resin, while the input power of less than 200 W for the SiC_w loading of 15% by weight was required to effectively cure the resin as seen in Figure 3(c). Finally, Figure 3(d) exhibits the temperature rise curves as a function of SiC_w content compared at a constant microwave input power of 200 W. From the plot, the initial rate of temperature rise of the BEP893 molding compound increases with increasing of SiC_w loading. The phenomenon is attributed to an increase in dielectric constant of a material

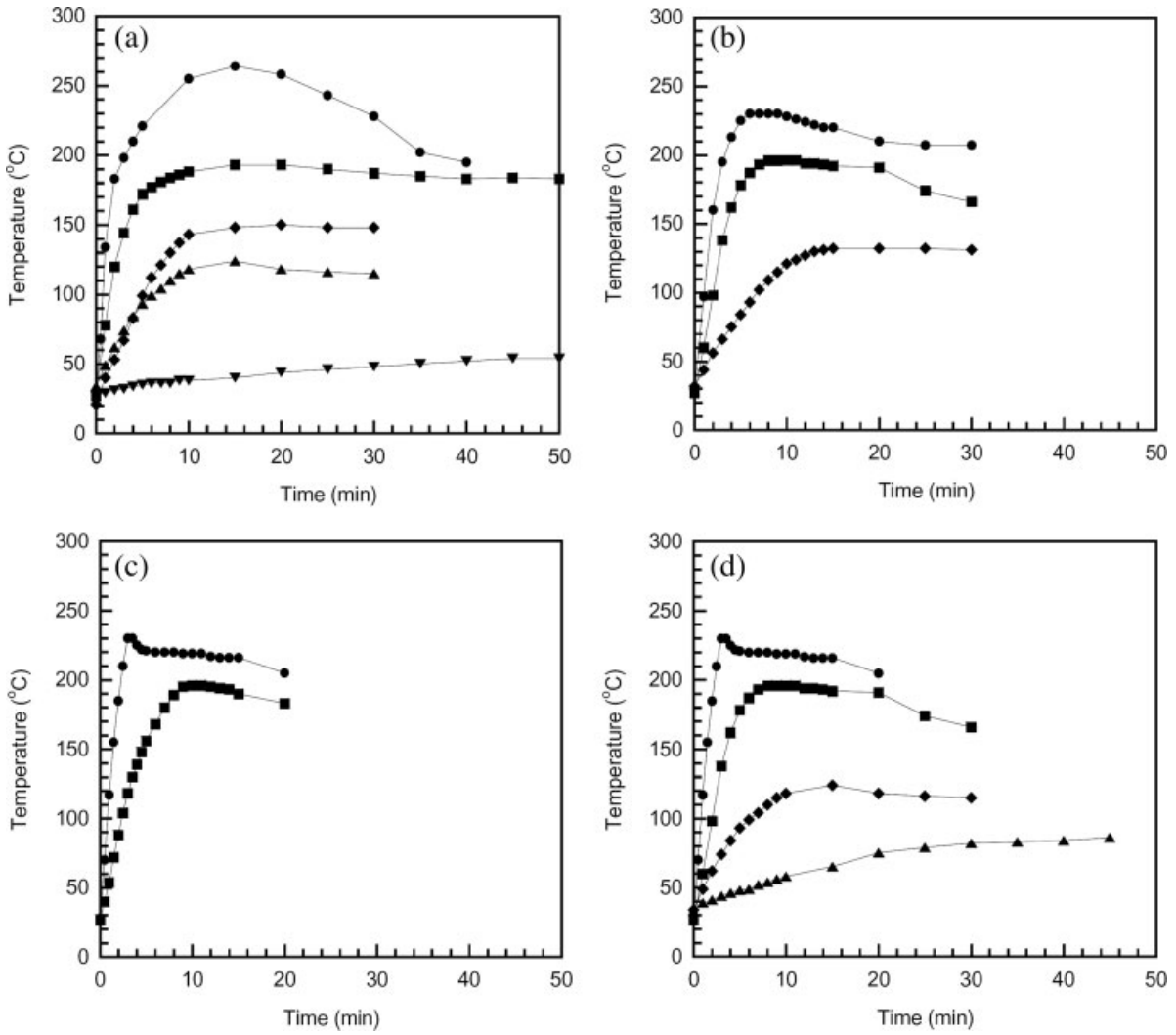

Figure 3 (a) Temperature rise curves of BEP893 molding compound at 5 wt % SiC_w loading: (●) 500 W, (■) 400 W, (◆) 300 W, (▲) 200 W, and (▼) 100 W. (b) Temperature rise curves of BEP893 molding compound at 10 wt % SiC_w loading: (●) 300 W, (■) 200 W, and (◆) 100 W. (c) Temperature rise curves of BEP893 molding compound at 15 wt % SiC_w loading: (●) 200 W, (■) 100 W. (d) Effect of SiC_w loading on temperature rise curves of BEP893 molding compound at a fixed microwave power of 200 W: (●) 15 wt %, (■) 10 wt %, (◆) 5 wt %, and (▲) 0 wt %.

with an incorporation of dielectric filler.[41] SiC_w is a ceramics with reported dielectric constant of up to 40[42] whereas the BEP 893 was measured to be about 4 as illustrated in Figure 4. In theory, the greater the dielectric constant of the material, the higher its microwave coupling ability will be. An addition of the SiC_w was found to systematically raise the dielectric constant of the BEP893 molding compound in a relatively linear manner as depicted in Figure 4, thus resulted in the enhanced microwave coupling of the compound with the filler loading.

Thermomechanical properties of conventional heat cured SiC_w-filled BEP893

The effect of SiC_w loading on dynamic mechanical properties of the fully cured BEP893 composites is shown in Figures 5 and 6. Figure 5 depicts the effect of the whisker loading (in the range of 0–20% by weight) on the storage modulus of the composites. From the plot, the presence of a high modulus SiC_w (400 GPa[28]) was found to significantly enhance both the glassy state modulus and the rubbery plateau modulus of the neat BEP893 matrix due to the reinforcing effect of the filler. Furthermore, the glass transition temperature (T_g) of the sample obtained from the peak position of the loss modulus as shown in Figure 6 was also found to increase with increasing the SiC_w content. The enhancement of the modulus and the T_g implies a relatively good adhesion between this filler and the BEP893 matrix.

There are many factors affecting a LCTE of a composite material including the ratio of filler in a poly-

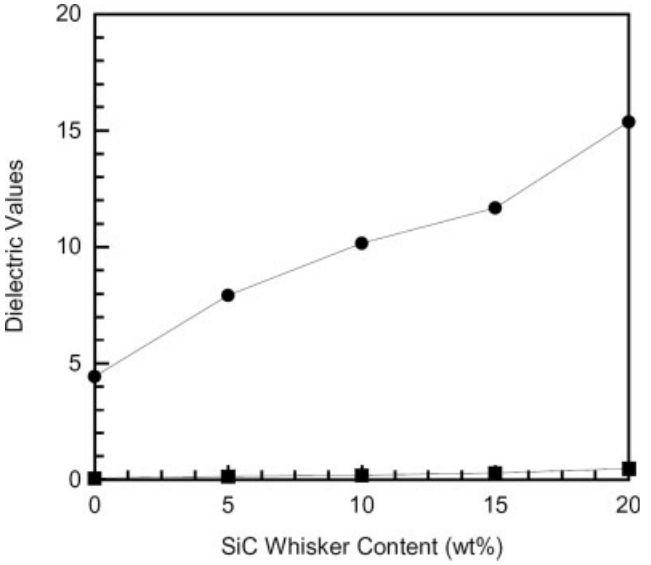

Figure 4 Permittivity and dielectric loss of BEP893 molding compounds as a function of SiC_w loading: (●) permittivity, (■) dielectric loss.

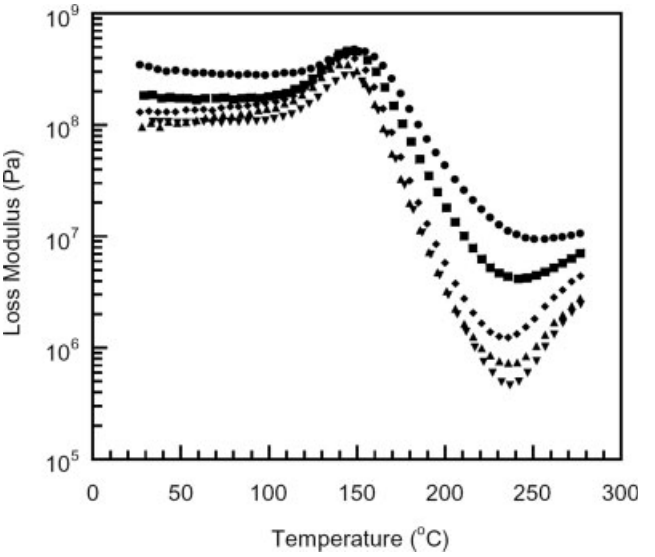

Figure 6 Effect of SiC_w loading on loss modulus of fully cured BEP893 composites: (●) 20 wt %, (■) 15 wt %, (◆) 10 wt %, (▲) 5 wt %, and (▼) 0 wt %.

mer matrix, LCTEs of the filler and the matrix, bond between the filler and the matrix, as well as the degree of polymerization of the resin.[43,44] Figure 7 presents LCTE of SiC_w-filled BEP893 at various contents of the filler. The figure evidently reveals that the LCTE systematically decreases with an increase in the SiC_w content. The reasons for this phenomenon are attributed to the adamantine nature of the whisker with a reported LCTE value as low as 4.7 ppm $^{\circ}C^{-1}$ (ref. 42) and the substantial interfacial interaction between the whisker and the matrix. From this figure, the LCTE value of the unfilled BEP893 was determined to be 64 ppm C^{-1} whereas those of the filled BEP893 are ranging from 51 ppm$^{\circ}$ C^{-1} at 5% by weight of SiC_w to 28 ppm $^{\circ}C^{-1}$ at 20% by weight of SiC_w. The SiC_w is, therefore, useful in lowering the LCTE of the BEP893 matrix. Low LCTE composite is required in some applications such as an electronic encapsulant. In our case, the SiC_w loading of 10% by weight in the BEP893 matrix was chosen for further investigation to compare the composite properties obtained by microwave cure and by

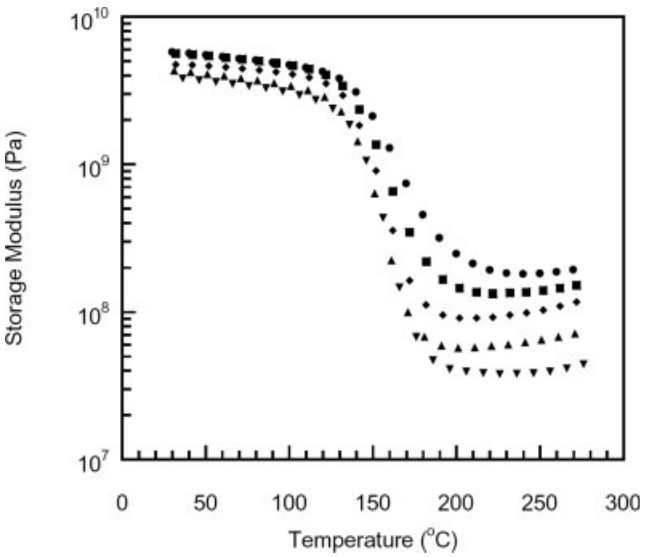

Figure 5 Effect of SiC_w loading on storage modulus of fully cured BEP893 composites: (●) 20 wt %, (■) 15 wt %, (◆) 10 wt %, (▲) 5 wt %, and (▼) 0 wt %.

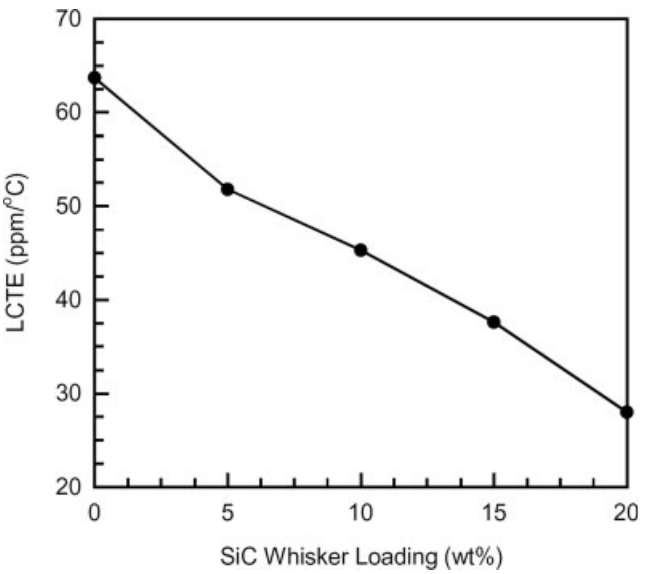

Figure 7 Linear coefficient of thermal expansion (LCTE) of SiC_w-filled BEP893.

conventional heat cure. The loading had been shown to provide sufficient microwave coupling as discussed in the previous section.

Comparison between microwave cured and conventional heat cured BEP893 composites

Figure 8 is the overlay plot of DSC thermograms of BEP893 molding compound at 10% by weight of the SiC_w cured isothermally at 200°C using a conventional oven. The graphs exhibit a systematic decrease in the exothermic curing peak as well as the evolution of T_g of the composite at different curing time. In addition, the peak exotherms were observed to slightly shift to higher temperature and the T_g expectedly increases with the degree of cure.

Figure 9 exhibits the DSC thermograms of the 10% by weight SiC_w-filled BEP893 after curing with microwave at 100, 200, and 300 W using constant cure time of 30 min. It can be clearly seen that the microwave input power significantly affects on the degree of cure of the BEP893 molding compound. Within 30 min, the input microwave power of 300 W was found to completely cure the BEP893 molding compound as signified by the disappearance of the exothermic peak of the DSC thermogram. Only partially cured samples, however, were obtained using 100 and 200 W of the input power. As a consequence, the microwave input power of 300 W was selected to process our molding compound. The optimal microwave irradiation time for curing the BEP893 molding compound using the input power of 300 W was also studied.

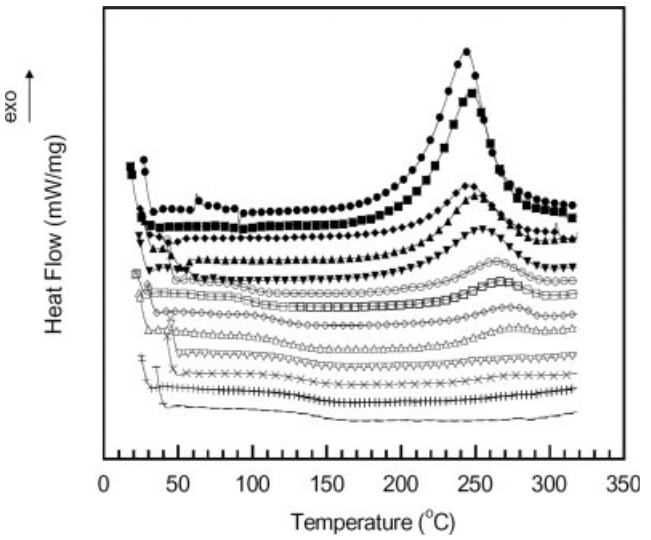

Figure 8 DSC thermograms at different cure time of BEP893 molding compound at 10% by weight of SiC_w: (●) 0 min, (■) 10 min, (◆) 20 min, (▲) 30 min, (▼) 40 min, (○) 50 min, (□) 60 min, (◇) 90 min, (△) 120 min, (▽ 150 min, (×) 180 min, (+) 240 min, and (−) 340 min.

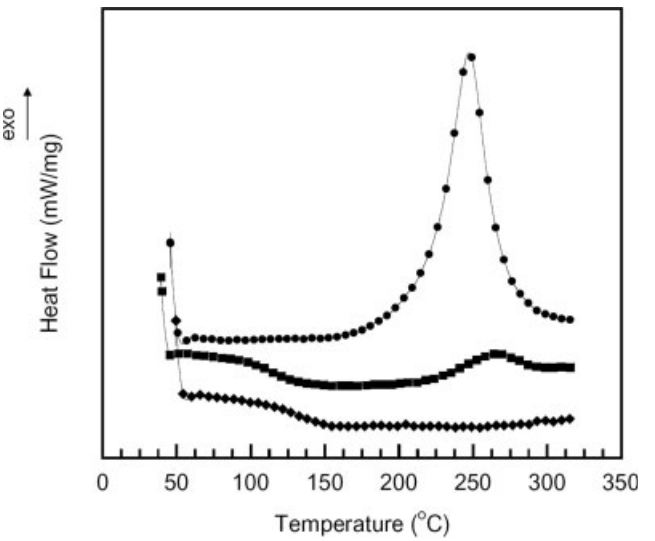

Figure 9 DSC thermograms of 10% by weight of SiC_w-filled BEP893 composites at different microwave powers (irradiation time = 30 min): (●) 100 W, (■) 200 W, and (◆) 300 W.

Figure 10 shows the effect of irradiation time on the conversion of the BEP893 molding compound filled with 10% by weight of SiC_w using a microwave input power of 300 W. The variable cure time of up to 90 min was examined and the cured samples were taken for DSC analysis. From the thermograms, the curing exotherm of the BEP893 molding compound was observed to be drastically reduced even when the cure time of only 5 min was used. The conversion-time diagram of the microwave heated BEP893 molding compound at 300 W and its

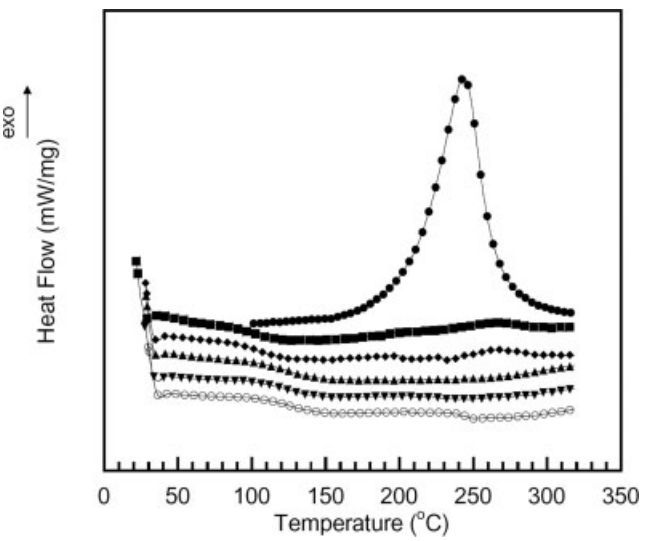

Figure 10 Effect of microwave irradiation time on conversion of 10% by weight of SiC_w-filled BEP893 molding compound at irradiation power = 300 W: (●) 0 min, (■) 5 min, (◆) 10 min, (▲) 20 min, (▼) 30 min, and (○) 90 min.

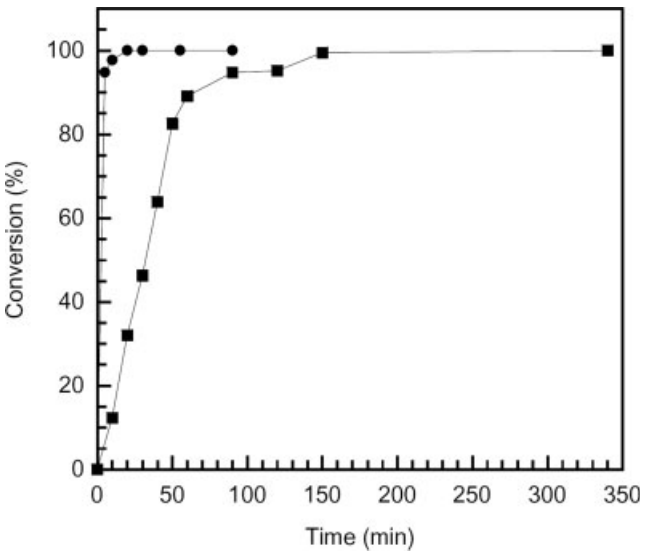

Figure 11 Conversion-time diagram of BEP893 filled with 10% by weight of SiC_w: (●) microwave cured at 300 W, (■) heat cured at 200°C.

conventional heat cured sample at 200°C is shown in Figure 11.

The conversion of each molding compound was calculated from the following equation.

$$\% \text{ conversion } (\alpha) = \frac{(\Delta H_0 - \Delta H)}{\Delta H_0} \quad (1)$$

where ΔH_0 is the curing enthalpy of the starting resin and ΔH is the curing enthalpy of the partially cured sample. Both enthalpies were determined from area under the exotherm in the DSC curve.[20,45]

Figure 11 evidently reveals the main advantage of using microwave technique to accelerate the curing process of the molding compound. From the plot, the curing time of 5 min at the microwave power of 300 W renders the molding compound with about 95% degree of conversion while the curing time of 10 min can provide a degree of conversion of nearly 100%. In the case of a heat cured sample at 200°C, the heating time of at least 150 min was necessary for the same sample to achieve the same degree of conversion. The potential energy saving from a substantial decrease in the processing time utilizing microwave irradiation was, thus, obtained.

The T_g of the BEP893 composites also increases in accordance with the degree of cure. The corresponding T_g-conversion curves of the above microwave cured and conventional heat cured samples, i.e., the systems shown in Figures 8 and 10, are also plotted in Figure 12. The curve shows the development of T_g as a function of percent conversion revealing the trend typically observed in several thermosets.[46–48] In the figure, slightly lower T_g values of the microwave cured samples than those of the conventional heat cured samples were observed. The different heating mechanisms i.e., by conduction throughout the whole sample in a conventionally cured sample and a sporadic heating from the surface of tiny SiC_w as well as the inside-out heating mechanism of the microwave during the processing may be responsible for the observed discrepancy.

Figure 13 depicts the dynamic mechanical characteristics of the fully cured BEP893 composites processed by microwave radiation. It was observed that the storage modulus at room temperature of the composite was about 5.5 GPa which is comparable

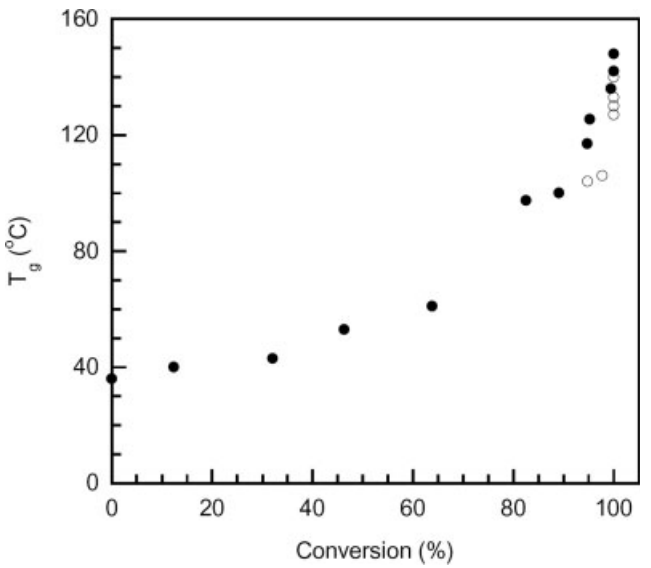

Figure 12 Glass transition as a function of cure conversion of 10 by weight SiC_w-filled BEP893: (○) microwave cured at 300 W, (●) heat cured at 200°C.

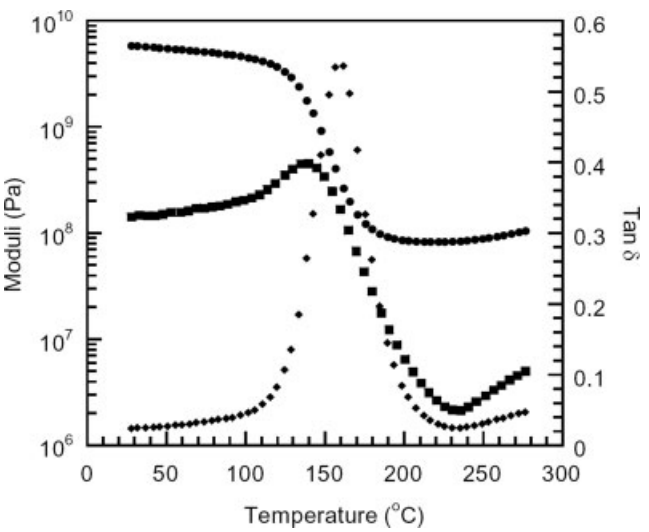

Figure 13 Dynamic mechanical properties of microwave-treated BEP893 composite: (●) storage modulus, (■) loss modulus, and (◆) tan δ.

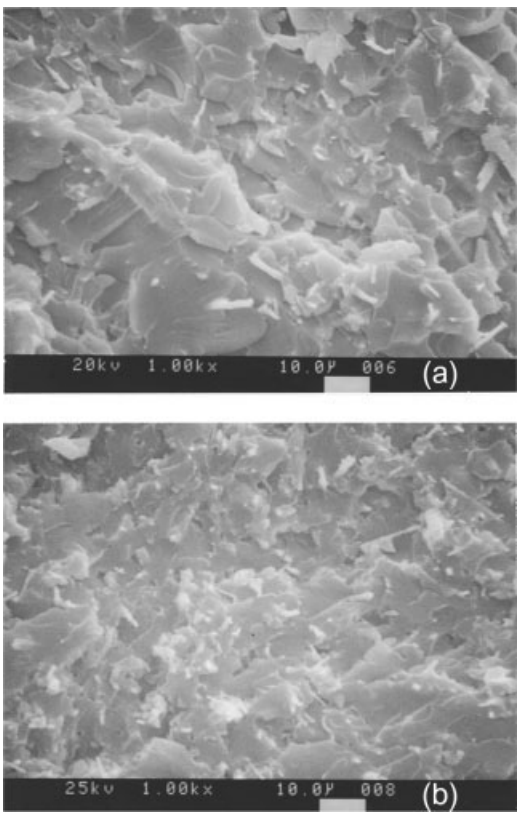

Figure 14 SEM micrographs showing fracture surface of BEP893 composites: (a) microwave cured, (b) heat cured.

to the value of 5.1 GPa of the BEP893 composite conventionally cured shown in Figure 5. The $T_{g,\mathrm{DMA}}$ of the microwave cured sample was determined to be 140°C which is slightly lower than the value of 145°C of the conventional heat cured sample shown in Figure 6. This phenomenon is consistent with the DSC result explained in the previous section.

SEM micrographs of the fracture surface of BEP893 composites cured by microwave irradiation and heat are shown in Figure 14. The micrographs reveal similarity in fracture surface morphology of the microwave cured and the conventional heat cured composite. In both cases, the fracture surfaces show substantial adhesion between the whisker and the matrix as seen from the tight interfaces between the two components with relatively short whisker pull-out length. The good interfacial adhesion of the filler and the matrix also explains the enhancement in dynamic mechanical properties and glass transition temperature of the obtained composites.

CONCLUSIONS

Microwave processing ability and thermomechanical properties of SiC_w-filled BEP893 were investigated. SiC_w was observed to effectively assist the microwave processability of the BEP893 resin. Optimal amount of SiC_w of 10% by weight at relatively low microwave power input of 300 W was found to sufficiently cure the BEP893 resin using a curing time of about 10 min. Microwave cured sample yielded slightly lower T_g than the conventionally cured sample. The different heating mechanisms of the conventional heat-cured and the microwave-cured samples are thought to be responsible for the discrepancies. SEM pictures of the composite fracture surface revealed substantial interfacial adhesion between the BEP893 matrix and the whisker.

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