Microwave processing of SiC nanoparticles infused polymer composites: Comparison of thermal and mechanical properties

Md. Reza-E-Rabby, Shaik Jeelani, Vijaya K. Rangari

Department of Material Science and Engineering, Tuskegee University, Tuskegee, Alabama 36088
Correspondence to: V. K. Rangari (E-mail: rangariv@mytu.tuskegee.edu)

ABSTRACT: The goal of this study is to compare thermal and mechanical properties of an epoxy resin system reinforced with SiC nanoparticles using both conventional thermal curing and microwave irradiation techniques. The microwave curing technique has shown potential benefits in processing polymeric nanocomposites by reducing the curing time without compromising the thermo-mechanical performances of the materials. It was observed from this investigation that, the curing time was drastically reduced to ~30 min for microwave curing instead of 12 h room temperature curing with additional 6 h post curing at 75°C. Ductile behavior was more pronounced for microwave curing technique while thermal curing showed brittle like behavior as revealed from flexural test. The maximum strain to failure was increased by 25–40% for microwave-cured nanocomposites over the room temperature cured nanocomposites for the same loading of nanofillers. The glass transition temperature (T_g) also increased by ~14°C while curing under microwave irradiation. © 2014 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2015**, *132*, 41708.

INTRODUCTION

Silicon carbide (SiC) is one of the ceramic fillers which are extensively used because some of its inherent characteristics such as high heat- impact- radiation- and oxidation resistance, high chemical stability, catalyst support, strengthening and increasing super plasticity of materials, wear resistance, etc.[1,2] These intrinsic properties facilitate SiC nanoparticles applicable in the field of semiconductor materials, optic devices, hard disk support from multichip modules, integrated circuit substrates, additive of space-flight product, catalysts, high precision machine, mirror or coating to protect from extreme ultraviolet environment, high temperature fluid transport parts, polishing media, hard coatings, and first-wall materials for fusion reactors. Numerous researchers investigated the compatibility of SiC nanoparticles into composites and have shown remarkable improvement in response to thermal and mechanical properties.[1,3–6] The efficiency and effectiveness of the nanofiller-matrix stress transfer mostly dependent on the chemical compatibility between these two components. The morphological investigation of SiC nanoparticles has revealed that, SiC nanoparticles have tendency to be oxidized with the formation of crystalline silicon dioxide (SiO_2) on the surface.[4] Therefore, a bridging network by strong chemical bonding between polymer and SiC can be established by in-situ functionalizing SiC in the presence of the hydrolyzed –OH group using the surface initiated polymerization (SIP) method and enhancement in tensile modulus (71%) and strength (166%) as well as improvement in thermal stability have been reported with the addition of SiC nanofillers.[4] The main challenges for inorganic SiC nanoparticles into polymeric composite system are the dispersion stability and compatibility for obtaining strong interfacial adhesion. The ultrasonic irradiation was found to be an efficient method for better dispersion of nanofiller into organic media.[3,6] The matrix system with 1.5 wt % nano-SiC infused into satin woven carbon fibers by vacuum assisted resin transfer molding (VARTM) method confirmed the sign of improvement in thermal stability and mechanical properties as well.[3] It was believed that the mechanism of such type of improvement by infusing nanoparticles into matrix media is because of involvement of some chemistry like inter-atomic or molecular activities, enhancement in crystallinity or cross linking of thermoset polymers.[3] The weight percentage of nanoparticles into epoxy resin also dictates the mechanical properties of nanocomposites. It has been observed that with the introduction of higher wt % of SiC nanoparticles into epoxy system, the mechanical properties are decreased albeit the wear resistance of nanocomposites are increased at higher concentration which is reasonable.[4,5]

In manufacturing of thermoset polymer nanocomposites, the curing is concluding phase, which drive the polymeric reactions by the process of cross-linking and finally solidified or hardened materials is obtained. Therefore, the degree of curing plays a

significant role in the properties of thermoset polymers. Additionally, post curing of polymers is also important for attaining maximum benefit in terms of mechanical, thermal or electrical properties. Generally conventional thermal cure technique consume more time that might hinder manufacturing rapidity. Therefore, the expedition has taken researchers to the alternate rapid curing techniques using UV rays,[7] Gamma rays,[8] electron beam,[9] and microwave irradiation.[7,9–11] However, microwave offers advantageous effect on polymers curing because of its selective heating with faster and effective curing technique compared to other methods of curing and numerous researchers have devoted to investigating this technique for synthesis and manufacturing polymer nanocomposites.[7,11–26] Microwave curing resulted in shorter curing time with higher strain to failure of the composites, improving the exfoliation of epoxy nanocomposite without compromising the mechanical properties of nanocomposites.[13,19–21,25,26] The glass transition temperature (T_g) and thermal stability were also appeared to be higher for microwave cured nanocomposites compared to conventional thermal curing.[19–21,23,27] The reason of enhancement in T_g by microwave curing is unrevealed until now; however, it is sometimes claimed that high heat input and heat transfer might affect polymerization process thereby causing more compact three-dimensional polymer chain network.[28] The presence of functional group might result localized superheating of the functional group could be the main mechanism of reaction rate enhancement of microwave energy.[26] Previously, it was hypothesized that the presence of microwave absorbent materials weakens the localized superheating when microwave is incident because of absorbing microwave energy during the curing process and the curing rate is required to decrease with higher loading of nanoparticles into matrix. It is well known that carbon nanotube/nanofiber, SiC, etc. are excellent microwave absorbents and when microwave was incident onto these nanoparticles, a number of physical changes were observed in terms of permittivity, light emission, structural reconstruction, spin resonance, etc.[16,18,29] It has been evaluated from a pyrometer study that, the release of extreme heat from single-walled carbon nanotube can increase temperatures close to 2000°C when it is subjected to microwave irradiation.[16] Study has also shown that energy induced by microwave irradiation can successfully attach multi-walled carbon nanotube (MWCNT) to polyethylene terephthalate (PET), polycarbonate (PC) and polyimide (PI) substrates with an anisotropic alignment of MWCNT on the substrates.[24] This "microwave-welding" can be accomplished in 1–5 s of microwave irradiation. The bulk substrate was found to be unchanged, although high temperature was generated which may be because of short time of heat conduction during the attachment without damaging the chemical bonding of substrates. An interesting study on energy consumption during curing of epoxy based nanocomposites revealed that epoxy system with 0.5–1 wt % of CNT require 40–50% less energy than that required for net epoxy curing under microwave irradiation.[15] Moreover, the energy consumption for conventional cured nanophased composites was found to be 3.5–5 times higher than the energy consumption during microwave curing with subtle change in mechanical properties of the nanocomposites.[15] In a nut shell it can be said that, microwave irradiation

technique has shown some promises for curing epoxy nanocomposites as ascertained in the open literature. Therefore, this research is another attempt to substantiate the sturdiness of this process in manufacturing polymeric nanocomposite. The present study focused on the effect of SiC nanoparticle on epoxy curing using microwave heating. Because the SiC nanoparticles are excellent absorbers of microwave energy (1300°C in 5 min of irradiation), we expected to cure the epoxy much faster and effectively than thermal curing.[30]

In this article, microwave curing of an epoxy based polymer nanocomposites reinforced with SiC nanoparticles was investigated. Comparison of thermal properties of the nanocomposites for microwave curing technique and traditional thermal curing technique were carried out using differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), dynamic mechanical analysis (DMA), and thermal mechanical analysis (TMA). Three-point bending test was also performed in order to evaluate mechanical properties of the microwave cured and conventional cured nanocomposite system. It was observed that, mechanical properties in terms of flexural modulus, strength and maximum strain to failure increased with 1 wt % loading of SiC while comparing room temperature cured nanocomposites. A dramatic reduction in curing time was observed while using microwave irradiation technique. Mechanical and thermal properties were found to be improved while using microwave curing technique.

MATERIALS AND METHODS

Materials

Spherical shape silicon carbide (β-SiC, 99.7% pure) nanoparticle (average particle size less than 30 nm) was purchased from MTI Corporation, USA. The specific surface area of SiC was greater than 80 m²/g. The TEM micrograph of as received SiC nanoparticles is shown in Figure 1. The matrix system in this study is a commercially available, low viscosity, two part system SC-780 epoxy resin purchased from Applied Poleramic (API), USA. This SC-780 toughened epoxy resin system was specifically developed for vacuum assisted resin transfer molding (VARTM) process. The recommended temperature for infusion is at 23–27°C. The curing can be performed at 25°C overnight subsequently post cured at 71–77°C for 6 h to achieve maximum mechanical and thermal properties.

Synthesis of Nanocomposites

The precalculated amount (0.5, 1, and 1.5 wt %) of nanoparticles (SiC) and part A of epoxy SC-780 was weighed carefully and mixed together in a 400-mL plastic beaker. The dispersion of SiC into Part-A epoxy was carried out using a high intensity ultrasonic irradiation of Sonic Vibra cell ultrasonic liquid process (Ti horn, 20 kHz, 100 W/cm²) for 60 min in a pulse mode of 20 s on and 10 s off at an amplitude of 35%. This time, pulse and amplitude were adjusted after several trials of mixing under ultrasonic irradiation. The entire mixing process was performed at 0°C by submerging the mixing beaker into an external cooling thermostat bath in order to avoid a temperature increase due to ultrasonic irradiation. It is noticed that sonochemical mixing produced a huge number of bubbles inside the nanofiller modified Part-A of epoxy resin. Therefore, the
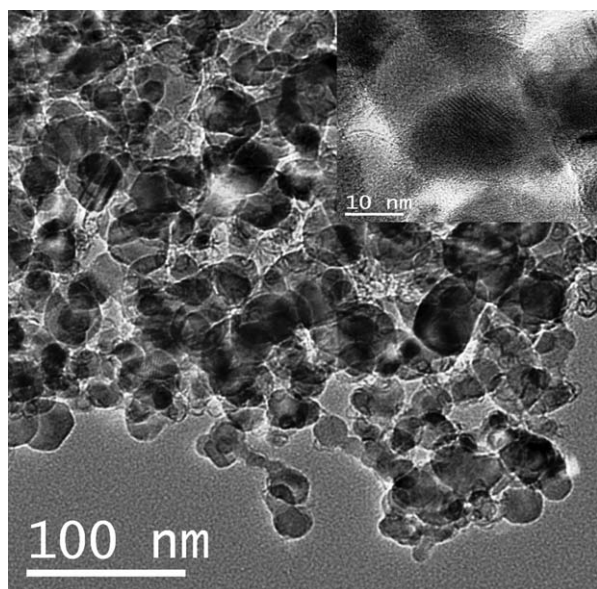
Figure 1. TEM micrograph of as received SiC nanoparticles. Inset image shows a high resolution image of the SiC lattice crystalline structure.

modified Part-A was kept in vacuum desiccators for 1 h to remove the entrapped voids. The part B which is an amine base hardener were added with the nano-modified Part A of SC-780 epoxy at a ratio of 100 : 22 and allowed to mix in a container specified for THINKY deforming mixer immediately after the degasification. THINKY hybrid deforming mixer ARE-250 was used for noncontact homogenous mixing of modified parts A and part B. In this technique the material container is set at 45° angle inside the mixer and revolves and rotates at high acceleration with the speed of 2000 rpm for 15 min. These dual centrifugal forces were given to the container that keep pressing materials to outward and down along with the slope of the inner wall of the container and accomplish powerful mixing and deaerating simultaneously. Afterward, the matrix system containing the nanoparticles was kept in desiccators for 15 min to remove the volatiles from the mixture. The matrix was placed in the steel flexure mold (according to ASTM standards) immediately after degasification. The specimens in the mold were allowed in a thermal oven at 25°C for 12 h, followed by a post curing at 75°C for 6 h to complete the curing cycle. For curing under microwave heating using a 2.45 GHz microwave processing oven BP210, the nanofiller infused epoxy matrix were transferred into Teflon pan and cured under microwave irradiation in two phases of curing cycle.

SiC are excellent microwaves absorbent which produces extremely high temperatures while absorbing microwave, resulting over burnt epoxy matrix system. Therefore, after a number of trials the procedure was set by splitting the steps of curing into two phases for microwave synthesis with the simple technique that is just placing Teflon pan with matrix into it. In first step mixture was heated using microwave irradiation at 20°C for 90 s and then paused process for 15 min at room temperature. In a second step at same temperature microwave irradiation was carried out for another 60 s and kept in the laboratory ambient condition 20 min for obtaining relatively superior quality microwave cured

specimens. These multistep heating cycles enable to reduce the generation of volatiles during the reaction. It was noticed that with the 1.5 wt % or higher loading of SiC in epoxy resin, microwave curing resulted a complete charred specimen due to high microwave absorbance by SiC. The microwave cured epoxy matrix samples were removed from the Teflon pan and the specimens for mechanical and thermal characterization were cut according to the standard specifications (Figure 2).

Characterization Techniques

Differential Scanning Calorimetry (DSC). Differential scanning calorimetry (DSC) experiments were carried out in an inert atmosphere using a METTLER DSC822^e supplied by METTLER Toledo. The temperature was ramped from 25 to 180°C at a heating rate of 5°C per minute under nitrogen atmosphere. Specimens were kept in aluminum pans and every time ~15 mg was taken for different category of specimen.

Thermogravimetric Analysis (TGA). To observe the decomposition temperature, residual solvent level, moisture content and the amount of inorganic fillers in polymers, the thermogravimetric analysis was carried out using a METTLER TGA/STDADSC851^e supplied by Mettler Toledo. The specimens were heated from 30 to 800°C at a ramp rate of 10°C per minute in an inert atmosphere of nitrogen. In this technique, weight changes in the specimen were measured as a function of temperature. The specimens were cut in small pieces of ~10–20 mg and placed in ceramic pan for running TGA for each category specimens while running the TGA.

Dynamic Mechanical Analysis (DMA). The storage modulus, glass transition temperature and cross linking density of the fully cured specimens were obtained using TA instrument Q800 series DMA. The test was run in a single cantilever mode with the amplitude of 15 µm, oscillatory frequency of 1 Hz and heating rate of 5° per minute with the temperature ranged from 30 to 180°C. The test was carried out according to ASTM
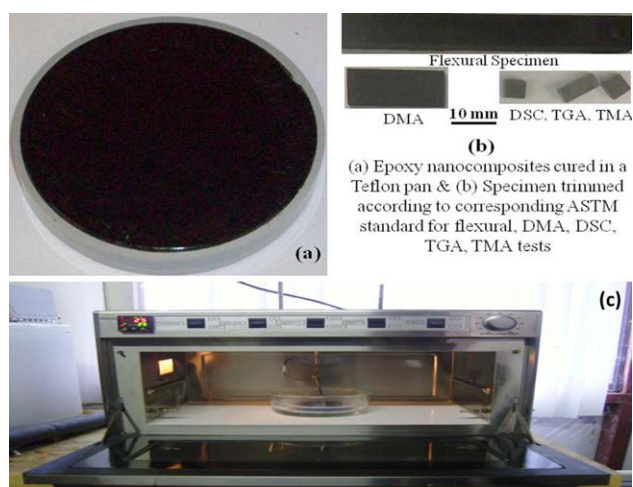

Figure 2. (a) As-prepared epoxy polymer cured in a Teflon pan using microwave irradiation, (b) samples cured precisely for flexural and thermomechanical testing, and (c) custom built BP-10 microwave. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
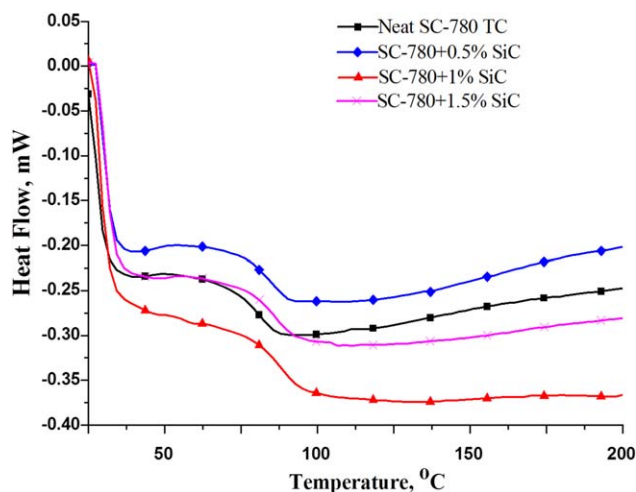
Figure 3. DSC graphs of traditional thermal cured epoxy nanocomposites with different loadings of SiC. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

D4065-01 with specimen dimensions of 25 mm in length, 3.25 mm in thickness, and 12.75 mm in width.

Thermal Mechanical Analysis (TMA). The thermal, mechanical analysis was carried out using a TA instrument Q400 series TMA for determining the coefficient of thermal expansion (CTE). The specimens were cut into small pieces of the approximate dimensions of $5 \times 5 \times 3$ mm^3 and dimension change was measured using the penetration probe of TMA instruments where the temperature was increased from 30 to 160°C with a ramp rate of 5°/min. From the test data, the ratio of dimensional change over temperature change was found. CTE was found using the formula as:

$$\alpha = \frac{1}{L} \times \frac{dL}{dT}$$

where, α is the CTE, the slope of the initial portion of the curves gives the value for dL/dT, and L is the thicknesses of the specimen.

Flexural Tests. The flexural tests were performed using a Zwick Roell instrument 2.5 kN load cell. The tests were performed according to ASTM D 790-07. The dimensions of the beam were ~4.5 mm in thickness direction, 12.5 mm width and 70 mm span length. A crosshead speed of 2 mm/min was used in the test machine. The support span to depth ratio (L/d) was 16–1 according to the ASTM standard. Five specimens were tested for each category of the specimens. This method was used to determine the flexural strength and modulus of elasticity of epoxy nanocomposites. The maximum stress at failure on the tension side of a flexural specimen is considered the flexural strength of the tested specimen.

Scanning Electron Microscope. The study of fracture surface as well as failure mode from the flexure test was carried out using JEOL JSM 5800 scanning electron microscope (SEM). The specimen was first placed on the specimen holder and sputtered with a very thin conductive layer of silver to prevent charge build-up by the electron absorbed by the specimen. A 5–20 kV accelerating voltage was applied to accomplish desired magnification.

RESULTS AND DISCUSSIONS

Differential Scanning Calorimetry (DSC)

Figure 3 depicts the DSC graphs of traditional thermal cured (TC) (at room temperature with additional post curing at 75°C) SC-780 epoxy resin neat and nanophased at different percentages of SiC loading. These graphs show that with the addition of SiC nanofiller, the endothermic peak shifted towards the higher temperature when compared with the neat SC-780 epoxy resin system indicating the increase in T_g of nanocomposites. These T_gs were determined as the inflection point of heat flow curve.[20,31,32] The T_g obtained from 1% and 1.5% loading of SiC in epoxy cured at room temperature were 88 and 87°C, respectively. These values are higher than the T_g for neat system (81°C) from the DSC analysis shown in Figure 3. It is well established that T_g increased with the increasing the cross-linking density and decreasing molecular mobility.[6] The addition of SiC nanoparticles up to an optimum level, imparts such contribution to enhancing the glass transition temperature of the nanocomposites. Figure 4 represents the DSC curves of microwave cured (MC) SiC infused SC-780 epoxy resin system at different loading which has a similar trend as for traditional cured resin system. T_g was also increased for SiC infused nanocomposites compared to neat epoxy. The measured T_g was found to be 89–91°C for neat and nanophased epoxy system which are much higher than those obtained from the traditional thermal cured nanocomposites. With higher loading (1.5%) of SiC, T_g is almost equal to that of neat SC-780 cured under microwave irradiation. This decrease is due to any of the following two reasons: (a) agglomeration or (b) effect of localized heating. As SiC absorbs microwave radiation, the localized heating caused overheating, resulting the burning of specimens and damaging bonding properties of matrix, finally decrease its mechanical and thermal properties. It was revealed from this DSC analysis that, microwave cured nanocomposites with 1% SiC infused SC-780 showed the highest glass transition temperature among all synthesized nanocomposites.
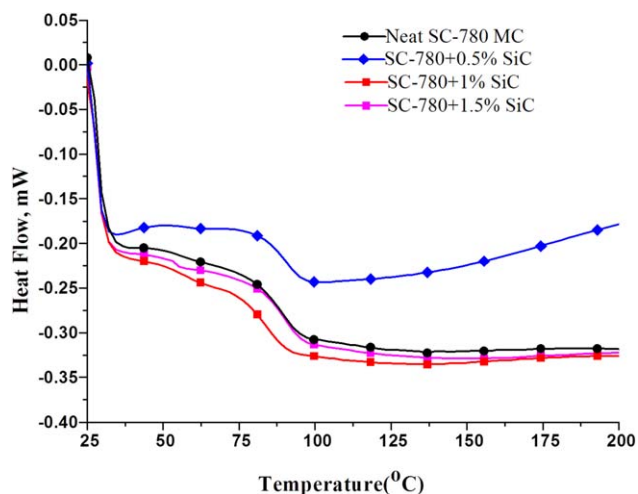

Figure 4. DSC graphs of microwave cured epoxy nanocomposites with different loadings of SiC. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
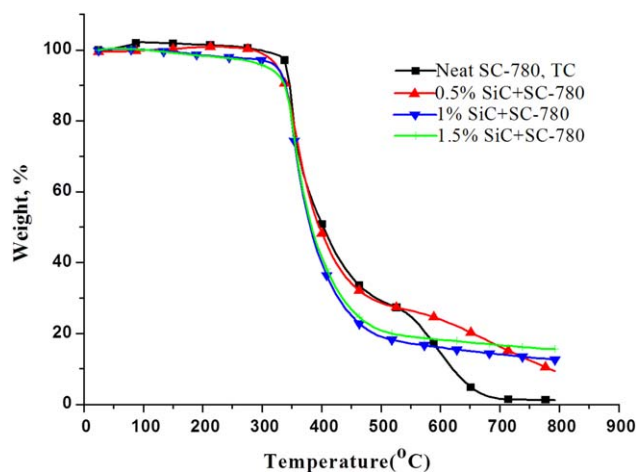
Figure 5. TGA graphs of thermally cured SC-780 epoxy with different loading of SiC. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
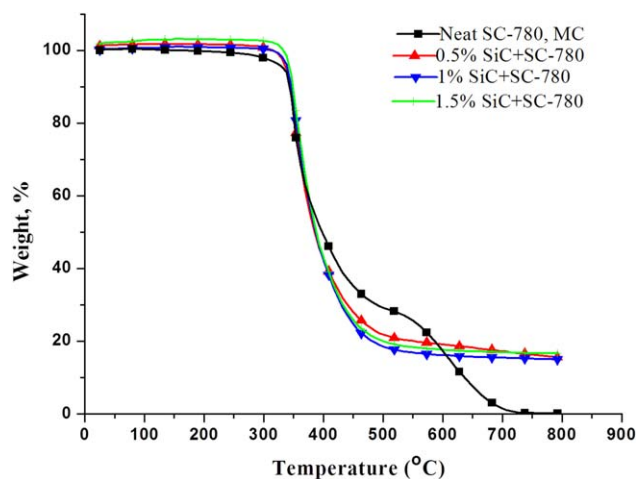

Figure 6. TGA graphs of microwave cured SC-780 epoxy with different loadings of SiC. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Thermogravimetric Analysis (TGA)

Figures 5 and 6 represent the TGA graphs of thermally cured and microwave cured neat SC-780 and nanophased epoxy resins with different loadings of SiC. In both curing techniques, the

neat SC-780 decomposes in two steps, at ~380°C (40% wt loss) and ~550°C (75% wt loss). This is because most of the volatiles were removed during the first stage of decompositions. For different loadings of SiC in nanocomposites, degradations were in
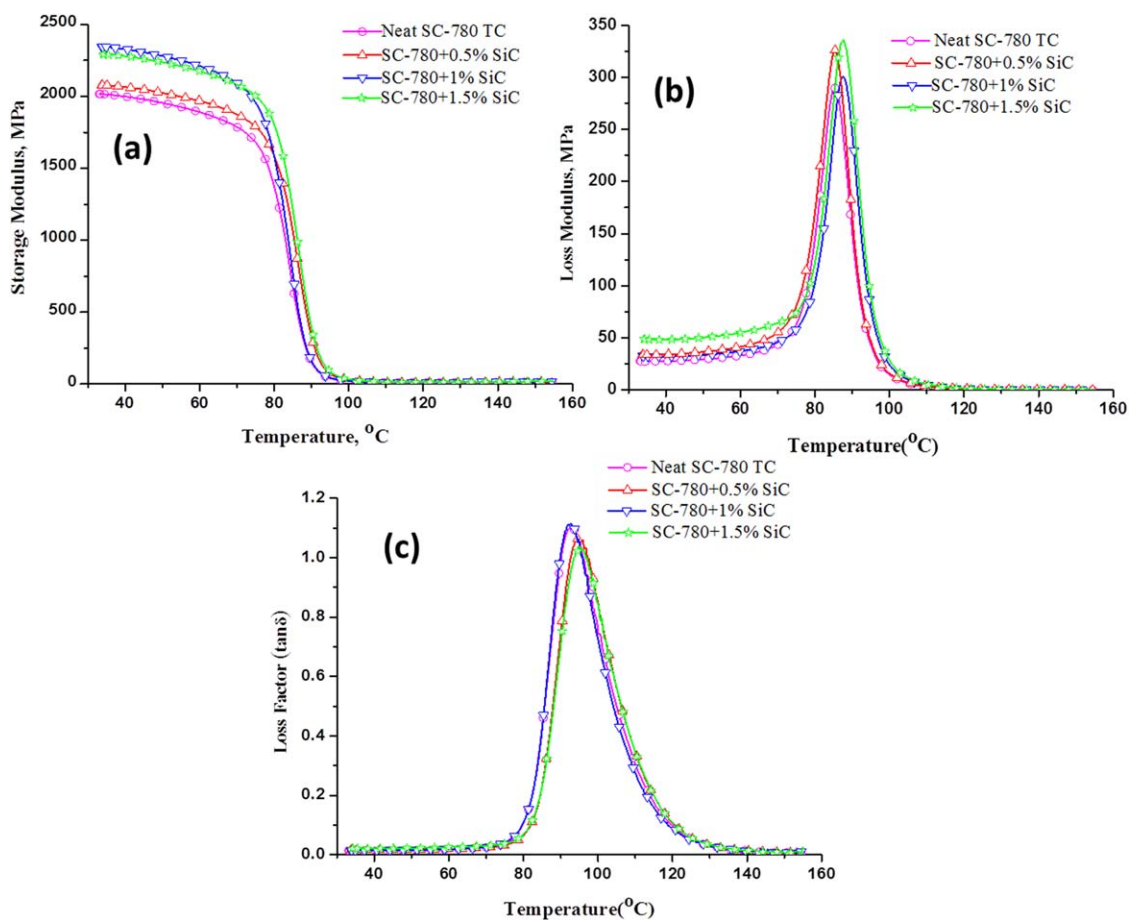

Figure 7. (a) Storage moduli of room temperature cured nanocomposites containing SiC at different percentages of loadings, (b) loss moduli of room temperature cured nanocomposites containing SiC at different percentage of loadings, and (c) loss factors of room temperature cured nanocomposites containing SiC at different percentages of loadings. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table I. Storage Moduli at 35°C and Glass Transition Temperature of Neat SC-780 with Different Loading of SiC for Room Temperature Curing

Specimen name	Storage moduli at 35°C (MPa)	Glass transition temperature in °C
Neat SC-780	2020 ± 2.5	85
SC-780+ 0.5% SiC	2081 ± 3.0	88
SC-780+ 1% SiC	2346 ± 1.5	86
SC-780+ 1.5% SiC	2292 ± 0.5	88

one step which was at ∼377°C suggesting that nanocomposites were cured completely in both traditional and microwave curing techniques.[20]

Dynamic Mechanical Analysis (DMA)

Dynamic mechanical analysis (DMA) over a wide range of temperature were performed to study the change in molecular structure and stiffness of the polymeric nanocomposites.[33] The glass transition temperature and yield information about the morphology of polymers are determined form DMA. The storage moduli, loss moduli and loss factor (tan δ) obtained from DMA tests are discussed with using Figures 7 and 8. Figure 7(a–c) depicts the (a) storage modulus, (b) loss modulus, and (c) loss factor as a function of temperature obtained from DMA

for room temperature cured neat SC-780 and nanophased epoxy containing SiC at different percentages of loading.

It was observed that the addition of SiC into thermoset polymers has some considerable effects on storage modulus in both glassy stage and rubbery stage depending upon the nanoparticle content in matrix. With 1% loading SiC, storage modulus was observed to be 2344 MPa at 35°C which is 16% higher than that of the neat system (2017 MPa). With the loading lower or higher than 1%, the storage modulus reduced as shown in Figure 7(b). This effect is due to the stiffness of SiC and the interfacial interaction along the areas between infused nanoparticles and polymer matrix. It was observed higher values for peaks with the higher loading of nanoparticles in nanocomposites. The values of loss moduli specify the conversion of heat energy which is used as a measurement of viscous component or unrecoverable oscillation energy dissipated per cycle.[34] Thus, it can be said that satisfactorily dispersed nanoparticles lend a hand to dissipate energy under visco-elastic deformation of surrounding matrix.

The glass transition temperatures (T_g) for different nanocomposites were obtained from peaks of loss moduli according to ASTM D 4065. T_g increases with higher loading of nanoparticle; this is because the mobility of polymers is reduced due to the presence of nanoparticles. However, the interfacial bonding are
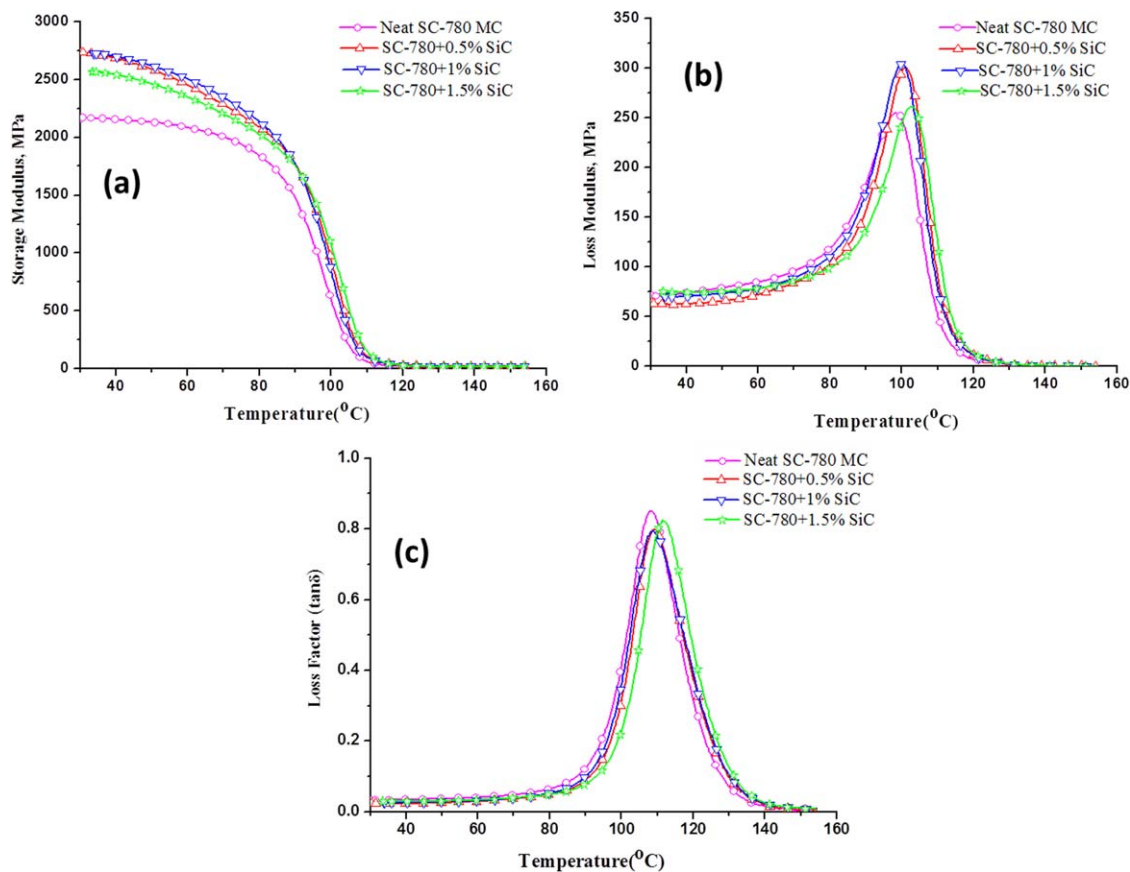

Figure 8. (a) Storage moduli of microwave cured nanocomposites containing SiC different percentage of loadings, (b) loss moduli of microwave cured nanocomposites containing SiC at different percentage of loadings, and (c) loss factor (tan δ) of microwave cured nanocomposites containing SiC at different percentages of loadings. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table II. Storage Moduli at 35°C and Glass Transition Temperature of Neat SC-780 and with All Loading of SiC for Microwave Cured Specimens

Specimen name	Storage moduli at 35°C (MPa)	Glass transition temperature in °C
Neat SC-780	2168 ± 1.5	99
SC-780+ 0.5% SiC	2820 ± 2.5	100
SC-780+ 1% SiC	2728 ± 2.0	100
SC-780+ 1.5% SiC	2566 ± 1.0	103

expected to be stronger between SiC and polymer which might contribute to enhanced T_g. Table I shows the storage modulus at 35°C and glass transition temperature for corresponding neat and nanocomposites with different nanoparticles infusion.

Figure 8 depict DMA graphs of microwave cured neat SC-780 and nanoinfused epoxy matrix system with SiC. Similar changing trend was shown for both storage modulus and loss modulus curves. However, the maximum storage modulus was observed for the nanocomposites with 0.5% SiC infusion that was 2818 MPa (30% enhancement compared to a neat system for microwave cured specimens) and the highest T_g from the peak of the loss modulus curve was 103°C for 1.5% SiC loading nanocomposites.

Interestingly, it was observed that microwave cured nanocomposites showed maximum of 16% improvement in glass transition temperature and up to 36% enhancement in storage modulus over room temperature cured nanocomposites regardless of the percentage loading of SiC. This increment is attributed to the microwave welding phenomenon where the epoxy surrounding the nanoparticles cures much better than other places of matrix nanocomposites as nanoparticles that absorb heat from the microwave, also might lead to faster curing.[24] Table II shows the storage modulus at 35°C and glass transition temperature for corresponding neat and nanocomposites with different nanoparticles infusion.

Thermo Mechanical Analyses (TMA)

Figure 9(a,b) depicts the dimension change with temperature of SC-780 epoxy resin system at different loadings of SiC for traditional thermally cured and microwave cured specimens. The CTE values were calculated from the slope of the TMA curves were shown in Tables III and IV. Two CTE from the same curve were obtained at glassy state (below T_g) and rubbery state (above T_g). The results showed that with the addition of nanoparticles into epoxy resin system, the CTE values decreased up to a certain level for glassy region. For rubbery region these values go up. Because during the heat transfer, the energy is stored initially by the nanoparticles at glassy region and beyond Tg, the initially stored energy by nanoparticles increases causing spread of atoms along the length of molecular chains. The decrease calculated up to 25% with 1.5% loading of SiC nanoparticles. These results match very well with literatures.[22]

It is interesting to note from TMA curve that, for almost all TMA examined nanocomposite, the expansion was inhibited or in some cases contracted with increasing temperature near the

glass transition temperature region except that for 1% SiC infused nanocomposites. The reason for this change in slope of CTE was attributed to the relaxation of residual stress–strain existing in the specimen around the glass transition.[22] It was observed that microwave cured specimens possess lower CTE values at glassy state comparable to room temperature cured nanocomposites and also showed the linear slope change at glass transition indicating good dimensional stability before glass transition temperature. However, at rubbery stage the CTE values for microwave-cured specimens were more compared with room temperature cured nanocomposites for 1% loading of SiC. Glass transition temperatures were found to increase over that of room temperature cured nanocomposite system. The reason for this enhancement is due to homogeneous heat flow from microwave irradiation with sporadic heating over the surface of the tiny nanoparticles as well as the inside-out heating mechanism instead of a convection heat transfer mechanism of traditional thermal curing technique.

Interestingly, no shape change was observed at the glass transition for the nanocomposites with 1% SiC cured under microwave irradiation as observed in Figure 9(b). This indicates that no
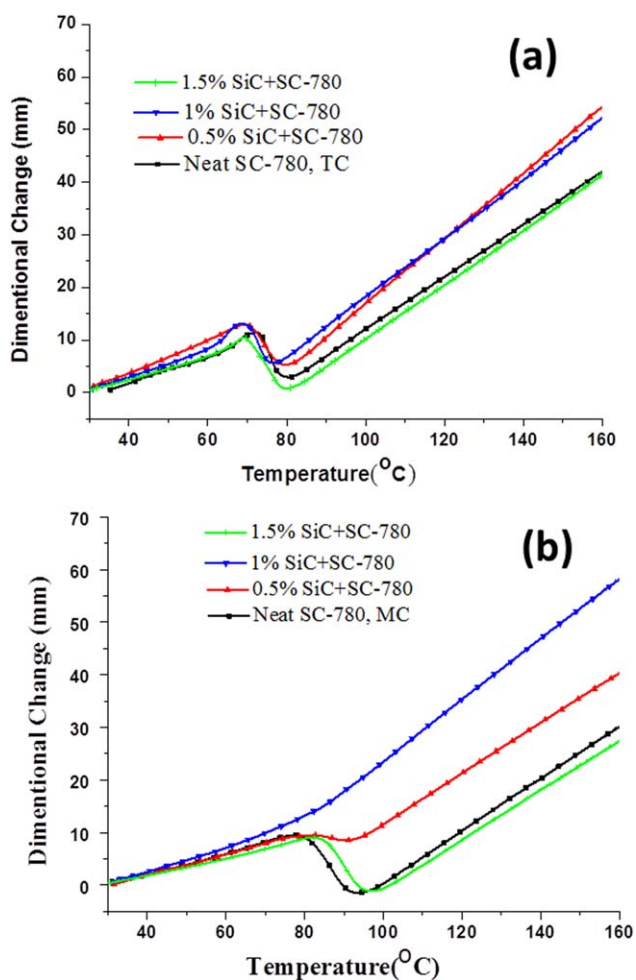

Figure 9. TMA curves of neat and nanophased SC-780 by (a) traditional thermal curing and (b) microwave curing techniques. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table III. TMA Data for Neat and Nanophased SC-780 of Thermally Cured Microwave Cured Nanocomposites

Specimen	Thermally cured		Microwave cured	
	CTE in glassy state μm/(m °C)	CTE in rubbery state μm/(m °C)	CTE in glassy state μm/(m °C)	CTE in rubbery state μm/(m °C)
Neat SC-780	94.6	168.4	79.1	187.7
SC-780+ 0.5% SiC	84.8	164.3	78.2	181.2
SC-780+ 1% SiC	74.9	160.6	75.1	178.7
SC-780+ 1.5% SiC	70.9	162.6	68.0	185.8

Table IV. Flexural Response of Neat and Nanofilled SC-780 Epoxy Cured at Room Temperature with Post Curing

Specimen ID	Ultimate flexural strength (MPa)	Average flexural modulus (GPa)	Average % strain at maximum force
Neat SC-780	107.7 ± 2.9	3.0 ± 0.1	4.9 ± 0.4
SC-780+0.5% SiC	113.8 ± 1.2	3.0 ± 0.0	5.1 ± 0.6
SC-780+1% SiC	120.6 ± 3.2	3.3 ± 0.2	5.0 ± 0.3
SC-780+1.5% SiC	115.2 ± 1.0	3.1 ± 0.1	5.3 ± 0.2

shrinkage was detected at this point. This characteristic of the curve suggests the proper exfoliation of nanoparticles into the epoxy matrix with good dispersion using microwave curing where molecular mobility decreased during glass transition and microwave welding phenomena was also observed.

Flexural Analysis

Figure 10 is the flexural graph of room temperature cured neat and nanophased SC-780 epoxy resin system at different percentages of SiC nanoparticles from 0.5 to 1.5%. From the graph, it is clear that with the addition of nanoparticles, the flexural strength and moduli of nanocomposites increased (gain) with respect to neat matrix. This is because of the decrease in mobility of the cross linking in polymers resulting better mechanical properties with an optimum level of nanofiller addition. Literatures have shown that higher loading of nanoparticles to matrix

system causes the nanocomposites to behave like brittle materials instead of ductile manner.[3,4,6] At 1% weight fraction of SiC the flexural strength and modulus were obtained at 120.6 MPa and 3.25 GPa, respectively. These values are ~10–12% higher (gain) than those of the neat system suggesting that SiC nanoparticles introduce additional mechanisms of failure and energy consumption without blocking matrix deformation.

The flexural graphs for microwave-cured nanocomposites are shown in Figure 11 for different weight fractions of SiC nanoparticles. This figure also shows the similar trend as described earlier stress, strain curve for conventional cured epoxy nanocomposites. For SiC loading the flexural strength and modulus (gain) increased up to 1% weight fraction. However, for 1.5% loading of SiC, both flexural strength and moduli decreased
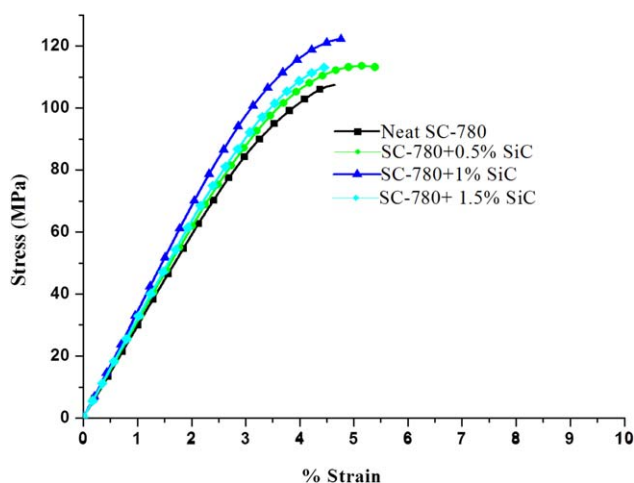

Figure 10. Stress–strain curve from flexural test for room temperature cured nanocomposites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
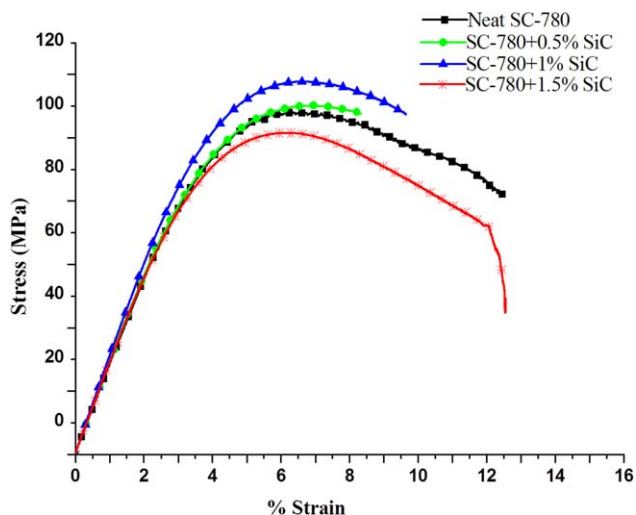

Figure 11. Stress–strain curves from flexural tests for microwave cured nanocomposites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table V. Flexural Response of Neat and Nanofilled SC-780 Epoxy Cured under Microwave Irradiation

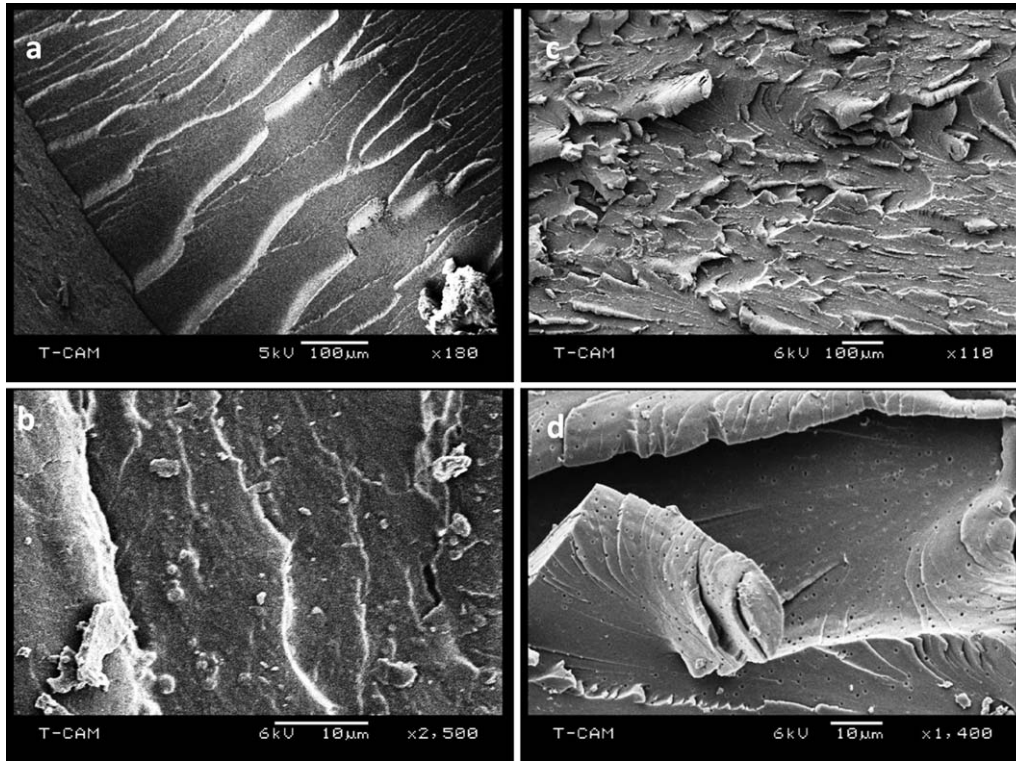| Specimen ID	Ultimate flexural strength (MPa)	Average flexure modulus (GPa)	Average % strain at maximum force
Neat SC-780	107.5 ± 4.7	2.8 ± 0.1	6.5 ± 0.1
SC-780+0.5% SiC	109.1 ± 7.6	2.9 ± 0.1	6.7 ± 0.3
SC-780+1% SiC	114.0 ± 9.8	3.0 ± 0.1	6.5 ± 0.3
SC-780+1.5% SiC	103.7 ± 11.8	2.7 ± 0.3	6.6 ± 0.4

Figure 12. SEM micrographs of fractured surfaces for SC-780 epoxy resin: (a,b) room temperature cured and (c,d) microwave cured nanocomposite.
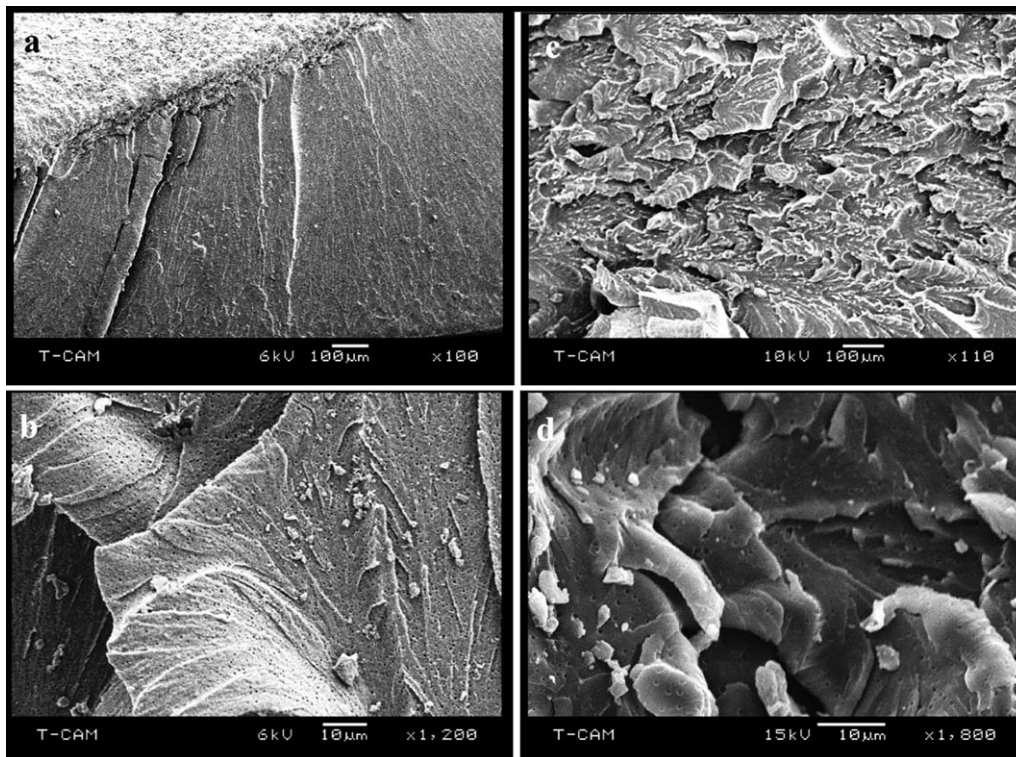

Figure 13. SEM micrographs of fractured surfaces for SC-780+1% SiC: (a,b) room temperature cured and (c,d) microwave cured nanocomposite.

(loss) because of over burning, soldering or over welding effect.[20,24]

Nanocomposites possess ductile behavior by microwave curing whereas traditional curing showed brittle behavior. These phenomena can be explained by microwave welding effect, where the matrix surrounded by nanoparticles cured much better than the far region where nanoparticles are not present. It was mentioned earlier that SiC is a good microwave absorbent and high heat is evolved when microwave is incident into it resulting better curing at the region of the nanoparticles and matrix interface. The over burning is due to the release of tremendous heat from the SiC nanoparticles when microwave is irradiated onto the matrix nanocomposites as it is well known that SiC is an excellent microwave absorbent. It was observed that the strain to failure was increased by ~40% for each percentage of loading of SiC nanoparticle in nanocomposites while curing is performed under microwave irradiation when compared with traditional curing. This increased strain is due to the endorsement in polymerization of epoxy resin by making the molecules more compact with increased density by microwave heating. Values obtained from flexural response are tabulated for both at room temperature cured and microwave cured nanocomposites in Tables IV and V. These results clearly indicates that the room temperature and microwave curing of epoxy in the presence of 1 wt % SiC nanoparticles shows a maximum increase in flexural strength and moduli. The higher than 1 wt % loadings of SiC nanoparticles in epoxy shows a decreased trend of flexural properties.

SEM micrographs were captured at the fracture surfaces of the failure plane form the flexural response. The micrographs for fracture surface from flexural tests are shown in Figures 12 and 13 for neat SC-780 epoxy resin system and SC-780 with 1% SiC both for traditional thermal cured and microwave cured specimens. These micrographs represent the cross-sectional view of the specimen, as one would look into either from left or right hand side of the specimen. The top surface of the specimen is under compression and the bottom is under tension.

In the neat system, it is evident that, initial crack formed at the edge of the tension side of the specimen. Relatively smooth surface was observed for the neat epoxy system of room temperature cured specimens as shown in Figure 12(a,b) indicating lower interfacial adhesion. However, for microwave cured neat SC-780, the fracture surface shown in Figure 12(c) was relatively rough which is expected in the sense that microwave irradiation causes enhanced polymerization by building more resin adhered to nanoparticles, consequently making the matrix more ductile. The magnified view of the matrix crack for the specimens cured under microwave irradiation shows lots of entrapped voids shown in Figure 12(d), which mostly is because of generation of volatiles during rapid reaction process with almost similar results from the mechanical response to flexural test. Similar phenomenon was also observed in the literature,[22] where there were evidence of formation of unexpected voids due to microwave curing technique that hamper minutely on mechanical properties compared to traditional thermal curing.

Figure 13 illustrates the fracture surface of nanocomposites with 1% loading of SiC. It was observed that for room temperature cured specimens the behavior of crack from in the radial direction from a center point at tension side of specimen and relatively rougher surface is seen upon the addition of 1% SiC into epoxy shown in Figure 13(a,b). These surfaces roughnesses indicate that the propagation of crack tip is distorted because of having SiC, which were intercalated into epoxy causing resistance to crack propagation. For microwave cured nanocomposites, the microwave welding phenomena were observed where the initial crack growth started vertically along the fracture surface from the tension side and then the crack split into two opposite directions shown in Figure 13(c,d) for its ultimate failure. However, voids were observed for microwave cured nanocomposites at higher magnification.

CONCLUSIONS

The investigations reveal successful features in producing polymeric nanocomposites with the infusion of nanofillers. With 1% loading of SiC into epoxy system, the flexural strength and modulus increased by 12 and 9.5%, respectively, over the neat epoxy with a minute enhancement in maximum strain to failure in case of room temperature cured nanocomposites. The microwave irradiation technique drastically reduced the cure time from 18 h to 30 min without compromising most of the thermal and mechanical properties by reducing the entrapment of un-cross-lined resin in the network. For microwave cured specimen, the maximum strain to failure were increased by 25–40% for 0.5% and 1% loading of SiC with respect to neat epoxy cured at room temperature without compromising the flexural strength and modulus. The glass transition temperature for room temperature cured nanocomposites was found to be increased by maximum 7°C from DSC results for 1% SiC loading. However, from DMA, the glass transition temperature was also increased by 10–16°C using a microwave curing technique compared with room temperature curing for corresponding concentration of nanofillers in the matrix. The storage modulus at 35°C was increased by 8–35% for each loading of nanoparticles into epoxy cured under microwave irradiation when compared with room temperature cured nanocomposites with specifically 35% enhancement for 1% SiC loading as observed from DMA. The CTE was decreased by 16% for microwave cured neat SC-780 epoxy resin in the glassy region compared to the room temperature cured specimens. Moreover, with increasing the amount nanoparticles into epoxy, CTE values are reduced as observed from TMA.

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