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Thermo-mechanical properties of silicon carbide-filled chopped glass fiber-reinforced epoxy composites

Gaurav Agarwal¹, Amar Patnaik^{2*} and Rajesh Kumar Sharma²

Abstract

The effect of addition of silicon carbide (SiC) filler in different weight percentages on physical properties, mechanical properties, and thermal properties of chopped glass fiber-reinforced epoxy composites has been investigated. Physical and mechanical properties, i.e., hardness, tensile strength, flexural strength, interlaminar shear strength, and impact strength, are determined with the change in filler content to notice the behavior of composite material subjected to loading. Thermo-mechanical properties of the material are measured with the help of a dynamic mechanical analyzer. The result shows that the physical and mechanical properties of SiC-filled glass fiber-reinforced epoxy composites are better than unfilled glass fiber-reinforced epoxy composites. Viscoelastic analysis for different compositions indicate that adding too much SiC content results in degradation in energy absorption capacity of the material and hence overall performance of the composites, whereas adding too much (more than 10 wt.%) SiC content increases the elastic behavior of the composite.

Keywords: Mechanical properties; Dynamic mechanical analyzer; SiC; Chopped glass fiber

Introduction

A test for mechanical properties is a prime area of interest. The mechanical properties of a material are those properties that involve a reaction to an applied load. The mechanical properties of a composite determine the range of usefulness of a material and establish the service life that can be expected. Most structural materials are anisotropic, which means that their mechanical properties vary with orientation (Shalin 1995). The variation in the properties can be due to the change in the microstructure of fiber/filler and matrix reinforcement. The materials can be subjected to many different loading scenarios, and the composite performance is dependent on the loading conditions. The common properties are considered to determine the loading constraints on the durability and service life effect tensile strength, flexural strength, hardness, interlaminar shear strength (ILSS), and impact strength (Rawlings 1999; Reid and Zhou 2000).

It is also necessary to determine the effect of change in temperature on the properties of fiber-reinforced epoxy composites because an increase in temperature results in the decrease in strength and ductility, whereas a decrease in temperature results in the increase in ductility and strength (Nielsen and Landel 1994). Apart from fiber-reinforced polymer composites, the composites made from both fiber/filler reinforcement performed well in many practical situations. Silicon carbide (SiC) filler particles were added to fiber-reinforced epoxy to enhance the mechanical properties of composites. A change in particle size, shape, and percentage content on the mechanical properties of fiber-reinforced polymer composites is an area of keen interest (Koh et al. 1993; Imanaka et al. 2001; Yamamoto et al. 2003). From research, it has been found that the structure and shape of silica particles have significant effects on the mechanical properties such as fatigue resistance, tensile, and fracture properties (Nakamura et al. 1991a, b; Nakamura et al. 1992).

The literature mentioned above gives a brief overview about the effects of addition of fiber/filler to mechanical and thermal properties of SiC-filled glass fiber-reinforced epoxy composites. It has been observed that various

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mechanical properties at optimum fiber loading conditions can be determined with the addition of fiber/filler reinforcement on matrix composites. To the author's knowledge, no work has been done to determine the optimum composition with the combination of fiber/filler and matrix to optimize the performance of the composite material.

Materials and methods

Material required

Chopped E-glass fiber (5 to 10 mm long, 200 G.S.M. density) manufactured by Ciba Geigy and locally supplied by Northern Polymers Ltd., New Delhi, India, is used as a fiber reinforcement. Commercially available SiC powder also known as carborundum of particle size 25 to 60 μm (density 2.6 g/cm^3) obtained from Silcarb Recrystallized Pvt. Ltd., Bangalore, India, is used as a particulate filler. The matrix material consists of epoxy resin LY 556 and room-temperature curing hardener HY951 supplied by Crystal Chemicals, Delhi, India. The composites were fabricated by blending epoxy resin, glass fiber, and SiC filler in a certain weight percentage reinforcement. Five different compositions of composites were prepared by varying the SiC filler reinforcement with fixed weight percentage (wt.%) of chopped glass fiber reinforcement. SiC filler in five different weight percentages (0, 5, 10, 15, and 20 wt.%) are added with fixed 20 wt.% of chopped glass fiber and remaining epoxy so as to notice the effect of SiC reinforcement on physical, mechanical, and thermal properties of chopped glass fiber-reinforced epoxy composites. The composites were prepared by blending certain weight percentage of fiber/filler and epoxy resin in separate containers and then poured in a mold of desired dimensions. Labeling was done with the help of rollers, and suitable weights were applied on top of the mold. Similarly, five different compositions of fiber, filler, and epoxy resin are poured in separate molds by varying five percentages of SiC filler content with that of the epoxy resin keeping the chopped glass fiber weight percentage as constant. The composites are then left for solidification at room temperature for 24 h. After the solidification process, the composites are then removed from the mold and marking is done as per the test standards. Specimens were prepared as per American Society for Testing Materials (ASTM) test standards for tensile, flexural, interlaminar shear strength, impact strength, and hardness tests.

Tests for mechanical properties

The mechanical properties were determined to understand the behavior of the material under different loading conditions. To understand how materials deform or break as a function of applied load, time, temperature, and other conditions, tensile strength, impact strength,

flexural strength, interlaminar shear strength, and hardness of the composite specimens were determined.

The capability of the composite material to resist abrasion or indentation is known as hardness. A Rockwell hardness tester is used to measure the hardness of the composite specimen. A spherical indenter (1/16 in or 1.5875 mm diameter) was forced into the surface of the material under conditions of controlled magnitude and rate of loading. The size of indentation is the measure of amount of hardness of the specimen.

Tests for mechanical properties, i.e., tensile test, interlaminar shear strength, and flexural strength, are done on a universal testing machine (UTM) Instron 1195. The tensile test is performed to measure the ductility of the material. Equal and opposite loads are applied at both ends of the rectangular specimen on the UTM Instron 1195 (ASTM 1976). Dimensions of the test specimen are 200 mm long, 11.5 mm wide, and with variable thickness (depending on the composition of the composite material). Flexural strength is the ability of the material to resist bending under the application of load. A three-point bend test was performed on the same UTM Instron 1195 with a span length of 30 mm in between the supports and a crosshead speed of 10 mm/min to measure the amount of failure when subjected to loading. Rectangular test specimen with dimensions 157 mm length, 12.7 mm width, and 6.35 mm thickness has been used for the experiment. Interlaminar shear strength tests are conducted as per ASTM D 2344-84 test standards on UTM Instron 1195 with span length of 50 mm and crosshead speed of 10 mm/min is maintained for testing (ASTM 1984).

Impact tests are done as per ASTM D 256 test standards (ASTM 1997) on plastic/composite specimen digital impact testing machine. Impact test is done to notice the energy consuming capacity of the composite material before fracture. Charpy V-notch impact test samples with dimensions 64 mm \times 12.7 mm \times 3.2 mm were prepared with a V-notch groove (2.5 mm depth) in the center of the specimen. The specimen was fixed on the impact tester such that the notch was at the opposite face to the striking end of the hammer.

Results and discussion

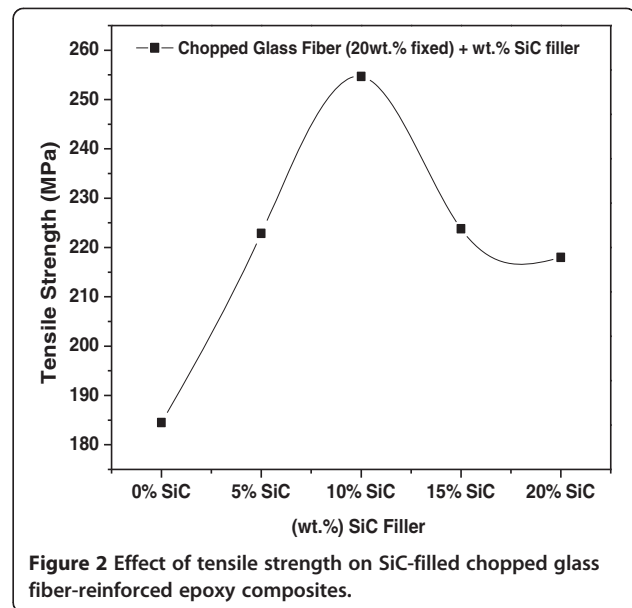
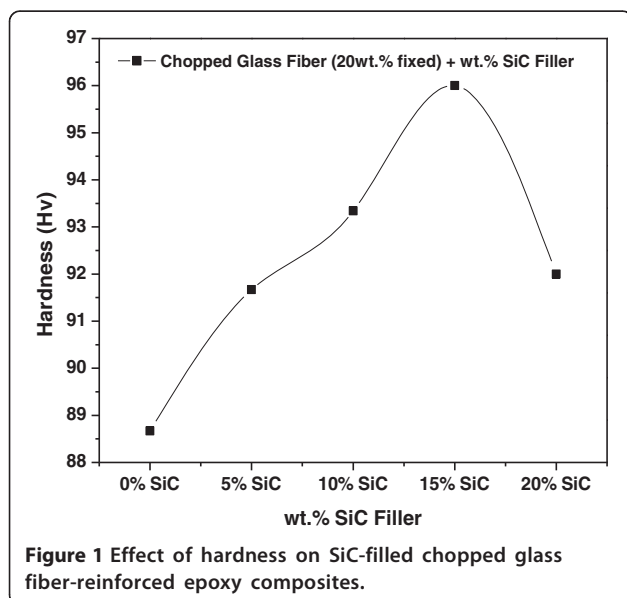
The physical and mechanical properties describe the behavior of the material to various practical applications. The properties such as hardness describe the physical state of the system. The mechanical property of the material is a measure of the behavior of the material under different loading conditions. Tests were done to notice the effect of addition of SiC filler on the physical and mechanical properties and the optimum fiber/filler loading of glass fiber-reinforced epoxy composites at which specific wear rate in minimum.

Effect of hardness on SiC-filled chopped glass fiber-reinforced epoxy composites

Hardness is a surface property and is a measure of wear resistance on the surface of the composite. The measured hardness values of all the five weight percentages of SiC reinforcement are shown in Figure 1. It can be seen that the hardness increases with the increase in the SiC content at a constant rate. Hardness increases linearly from 0 wt.% SiC content to 15 wt.% SiC content, but further increase in SiC content results in the decrease in the value of hardness. The increase in the value of hardness are attributed to the fact that as the density of composition increases due to filler particles introduced between the fiber and the matrix, the compression value of the specimen decreases and hardness increases. Addition of a small volume fraction of graphite, SiC, and alumina can significantly improve the hardness and wear resistance of the composites. With the addition of SiC filler content, the filler particles (SiC) fill in the gap between the fiber and the matrix and form a more dense structure and hence hardness increases (Mohamed et al. 2009).

Effect of tensile strength on SiC-filled chopped glass fiber-reinforced epoxy composites

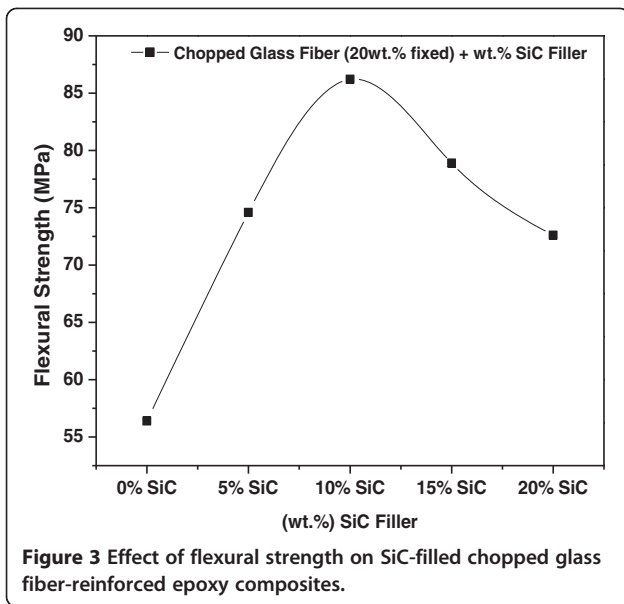
The ultimate tensile strength is an engineering value calculated by dividing the maximum load on a material by the initial cross section of the test specimen. Figure 2 shows the graph of tensile strength versus the SiC-filled chopped glass fiber-reinforced epoxy composite. Tensile strength increases from 0 to 10 wt.% SiC content, whereas further increase in SiC content results in the decrease in the value of tensile strength. This is because filler particles act as a barrier in



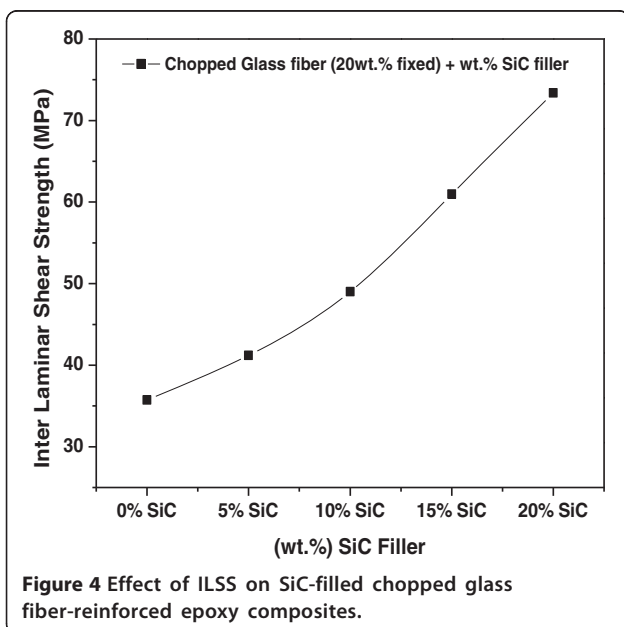
transferring stress from one point to another; and an increase in SiC content beyond 10 wt.% results in the increase of transfer of stresses from one point to another. Also as the fiber/filler content increases, the bonding surface area increases and hence bonding strength decreases. Due to insufficient amount of bonding between three different constituents, the loads may not effectively be transferred from one end to another and hence there is reduction in tensile strength of the composite. Tensile strength of carbon fiber-reinforced polyetheramide improves significantly with the addition in the percentage of fiber/filler reinforcement. The extent of improvement of tensile strength however is not proportional to the amount of carbon fiber reinforcement (Sua et al. 2005; Devendra and Rangaswamy 2012).

Effect of flexural strength and interlaminar shear strength on SiC-filled chopped glass fiber-reinforced epoxy composites

Figure 3 shows the flexural strength of SiC-filled glass fiber-reinforced epoxy composites. Flexural strength increases from 55 to 87 MPa with the increase in SiC filler content from 0 to 10 wt.%; an increase in the flexural strength beyond 10 wt.% SiC content results in the decrease in the value of SiC content. When the specimen is placed on two support points and load is applied from the top of the specimen, then the specimen is subjected to bending and the top layer is subjected to compressive loading, whereas the bottom layer is subjected to tensile loading. When the bonding between the fiber/filler and the matrix is increased, a flexural strength increases and strong bonding transfers loads from one end to another resulting in the increase in flexural strength of the specimen, whereas when the percentage of fiber/filler exceeds the required percentage, then the surface area increases,



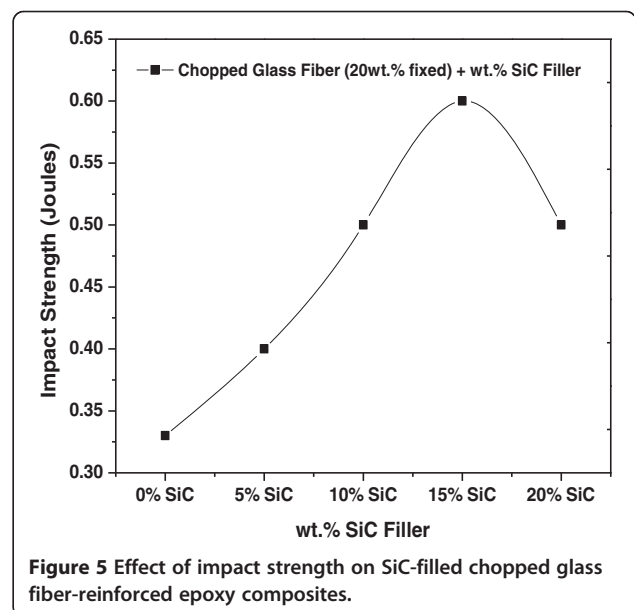
while the weight percentage of the matrix decreases; therefore, the bonding strength decreases, and loads cannot be effectively transferred from one part to another resulting in the decrease in the flexural strength of the composite. Another reason for the reduction in flexural strength with the addition of SiC filler is that the addition of SiC content beyond 10 wt.% disturbs the matrix continuity and reduction in bonding strength between filler, matrix, and fiber (Kaundal et al. 2012). Interlaminar shear strength is the measure of shear strength between the layers of fibers. Figure 4 shows the ILSS value of SiC-filled



glass fiber-reinforced epoxy composites. The ILSS value increases with the increase in the value of SiC content because SiC filler particles stick to the ends of fibers and act as a barrier in the transfer of shear stress from one part to another. A proportional graph is obtained between the weight percentage of SiC filler reinforcement and ILSS value, i.e., ILSS increases in equal proportion with the increase in the value of SiC content.

Effect of impact strength on SiC-filled chopped glass fiber reinforced epoxy composites

The material's resistance to fracture is known as toughness. It is often expressed in terms of the amount of energy a material can absorb before fracture. A ductile material can absorb a considerable amount of energy before fracture, while a brittle material absorbs very little. Figure 5 shows the increase in the value of impact strength with the increase in the percentage of fiber/filler reinforcement. The value of impact strength increases from 0 to 15 wt.% and further decreases for 20 wt.% SiC filler reinforcement. The increase in impact strength is due to the weaker bond, i.e., as the bond strength between the glass fiber, SiC filler, and the epoxy resin reduces, the impact energy absorbing capacity of the composite increases. Also, a large amount of energy is absorbed by the crack initiated along the fiber/filler/matrix interface during debonding. The energy absorbing capability of composites depends on the properties of the constituents. Based on the literature review, it has been found that the benefits of using SiC as reinforcement are improved stiffness, strength, and chemical stability (Kaundal et al. 2012).



Thermo-mechanical properties of composite (dynamic mechanical analysis)

A common cause of tribological failure of composite parts is the inability to support a given load due to the loss in modulus at frictionally induced elevated temperatures. Dynamic mechanical analysis (DMA) has emerged as one of the most powerful tools available for the study of the behavior of materials. Simply stated, DMA measures the viscoelastic properties of materials. DMA plot provides values of those temperature regions where material properties are very stable with temperature and where rapid changes will occur that would render the material useless (Ward and Sweeney 2004). One important application of DMA is measurement of the glass transition temperature of polymers. Amorphous polymers have different glass transition temperatures, above which the material will have rubbery properties instead of glassy behavior and the stiffness of the material will drop dramatically with an increase in viscosity. At the glass transition, the storage modulus decreases dramatically and the loss modulus reaches a maximum (Agarwal et al. 2013). DMA of chopped glass fiber SiC filler-reinforced epoxy composite have been carried out in this study to investigate the variation of storage modulus (E'), loss modulus (E'') and damping factor ($\tan \delta$) i.e.:

$$\tan \delta = \frac{E'}{E''} = \frac{\text{storage modulus}}{\text{loss modulus}} \quad (1)$$

Equation 1 represents that $\tan \delta$ is the ration of storage modulus to loss modulus, i.e., as the value of loss modulus increases, the value of storage modulus decreases (Agarwal et al. 2013). Similar trend is noticed in Figures 6 and 7. All tests run as per ASTM D4065-94 test standards using a fixed frequency of oscillation of 1 Hz and a sample heating rate of 2°C/min. All tests

were initiated from room temperature and goes to 200°C temperature. The mode of stress applied is flexure, and the fixture configuration is a single cantilever beam. Figures 6, 7, and 8 show the most common graphic representation for elastic or storage modulus, the viscous or loss modulus, and $\tan \delta$ as a function of temperature, respectively.

Figure 6 shows a graph of storage modulus versus temperature for SiC-filled chopped glass fiber-reinforced epoxy composite. Storage modulus values for chopped glass fiber SiC filler-reinforced epoxy composites show a linear trend with slight decrease in the value of storage modulus with the change in temperature (room temperature to 70°C). This shows glassy regime in region 1, i.e., from room temperature to 65°C. Region 2 shows a sharp decline in the values of storage modulus with the increase in the values of temperature (65°C to 90°C), i.e., the material changes from glassy to rubbery transition regime in the temperature range of 65°C to 90°C; due to a sharp drop in the value of storage modulus, the material has lost its usefulness as a structural material. Due to amorphous structure in the polymer and the presence of unorganized intermolecular structure, the storage modulus value drops suddenly. Region 3 again is a straight line showing negligible decrease in the values of storage modulus with the increase in the value of temperature. Here, the values of storage modulus nearly tend to zero, which shows a rubbery regime indicating degradation of the moduli above 90°C. In the glassy state, i.e., at low temperatures, stiffness is related to the small displacement of molecules, whereas in the rubbery state at high temperature, molecular chains have considerable flexibility; so that in the undeformed state, they can adopt conformations that lead to maximum entropy. The higher values in region 1 and sharp decrease in the values in region 2 are due to the fact that in region 1, the material is in glassy state in which the contribution of elastic modulus is more than the viscous modulus, whereas in region 2, the material is in glass transition stage in which a change from glass transition state into rubber elastic state takes place. In the glass transition region, i.e., from glassy to the rubbery transition regime region, the value of storage modulus falls during heating to a level of 1,000th to 10,000th of its original value (Jinmin et al. 1994).

Figure 7 clearly shows the value of loss modulus with the increase in the value of temperature. From the values obtained in the graph, we notice that the values of loss modulus rise to a maximum as the storage modulus values are in their most rapid rate of decent. The peak of the loss modulus curve denotes the glass transition temperature (T_g). Figure 7 denotes the glass transition temperature as 90°C and desired loss modulus values (minimum values) obtained for 10 to 20 wt.% SiC content. This may be due to the increase in the percentage of fiber/filler content, i.e., as the percentage of fiber/filler content increases, the height of the E'' curve decreases and the peak width spreads across a wider temperature

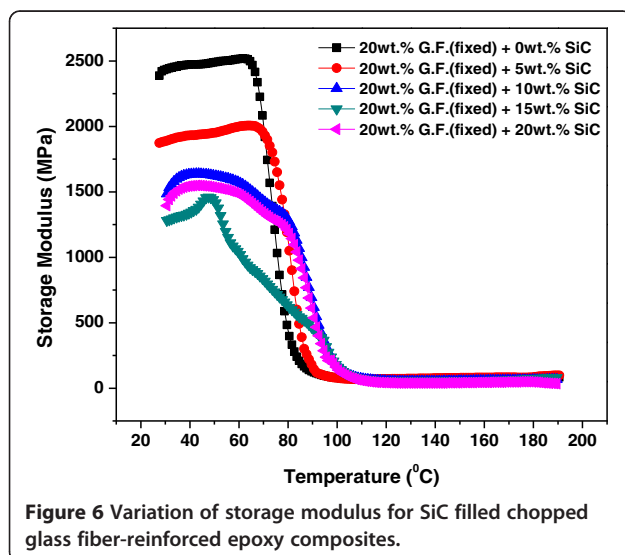


Figure 6 Variation of storage modulus for SiC filled chopped glass fiber-reinforced epoxy composites.

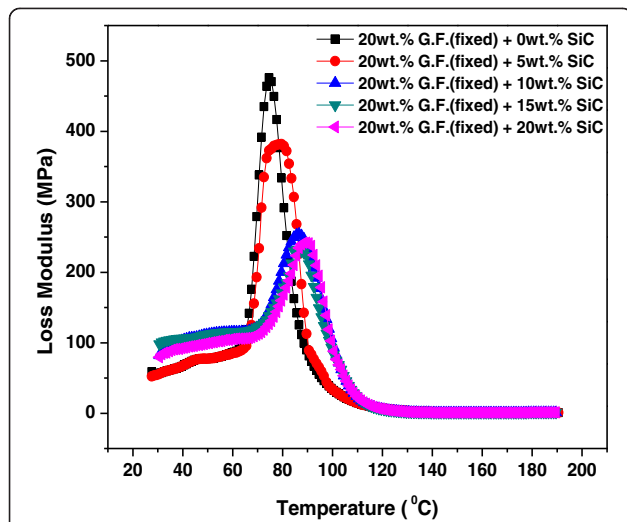


Figure 7 Variation of loss modulus for SiC filled chopped glass fiber-reinforced epoxy composites.

range leading to a decrease in the value of loss modulus (desired value obtained). Further, it has also been observed from Figure 7 that the composition with lower fiber/filler content peak of glass transition temperature shifted towards lower temperature with a sharp peak and higher value of loss modulus.

Figure 8 shows the graph of tan delta versus temperature. The tan delta curve closely resembles the loss modulus curve leading to the glass transition tan delta values well below 0.1. The rapid rise in tan delta values closely resembles the rapid decline in storage modulus values. The peak value of tan delta indicates that the material is nonelastic, whereas the lower value of tan delta indicates that the material is elastic in nature (Agarwal et al. 2013). Region 1 shows that the material is highly

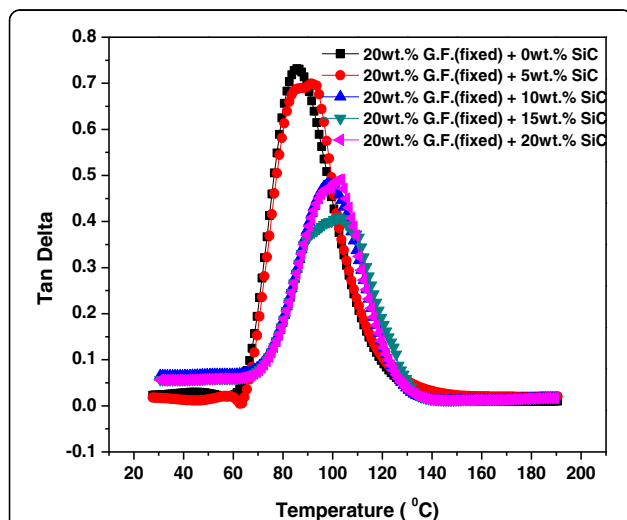


Figure 8 Variation of tan delta for SiC filled chopped glass fiber-reinforced epoxy composites.

elastic in this region, and as the value of tan delta shifts to region 2, the nature of material shifts from elastic to plastic. The peak value of tan delta indicates that any further increase in temperature beyond this changes the material from elastic to plastic zone. Figure 8 shows that as the percentage of fiber/filler increases, the peak value shifts towards higher temperature resulting in wider elastic range of temperature, whereas as the percentage of fiber/filler decreases, the peak value shifts towards the lower temperature range resulting in narrow elastic range.

Cole-Cole plot analysis

Figure 9 shows the analysis carried on SiC-filled chopped glass fiber-reinforced epoxy composites using Cole-Cole analysis. A Cole-Cole plot is used to predict the nature of a composite whether it be homogeneous or heterogeneous (Kumar et al. 2011). A Cole-Cole is plotted for storage modulus and loss modulus where the values of loss modulus ($\log E''$) are plotted on x -axis and the values of storage modulus ($\log E'$) are plotted on y -axis. Cole-Cole plot is used to describe the analysis of molecular architecture. Changes in the architecture of the model samples were readily detected as systematic variations in shape and displacements on Cole-Cole plot. A homogeneous system typically exhibits a semicircular curve, whereas a heterogeneous system typically exhibits an irregular curve (imperfect semicircle); also, the nature of curvature obtained in Cole-Cole curve determines the adhesion between fiber and matrix.

Investigated values of SiC-filled chopped glass fiber-reinforced epoxy composites represent imperfect semicircles and hence are heterogeneous in nature. Also, unfilled glass fiber-reinforced epoxy composites exhibit

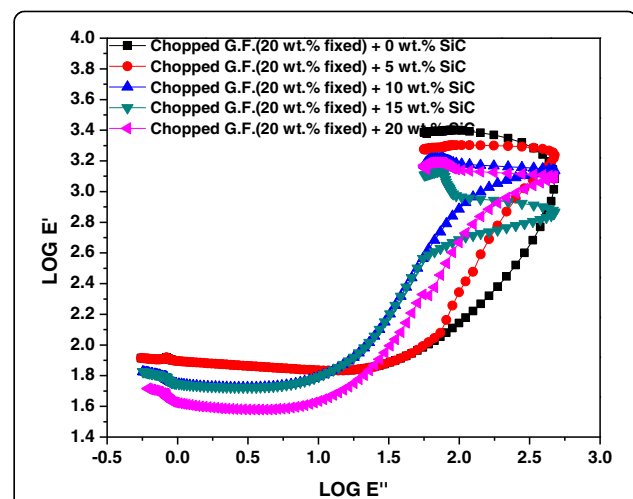


Figure 9 Cole-Cole plot for SiC-filled chopped glass fiber-reinforced epoxy composites.

better interfacial characteristics and some homogeneous nature with respect to SiC-filled glass fiber-reinforced epoxy composites. This is because adding SiC content breaks the matrix continuity, and the nature is shifted more towards heterogeneous tendency.

Conclusions

Experimental observations are carried out on SiC-filled chopped glass fiber-reinforced epoxy matrix composite to notice the effect of physical, mechanical, and thermal properties. Based on the experimental observations, the following conclusions can be drawn:

1. Mechanical properties such as hardness, tensile strength, interlaminar shear strength, flexural strength, and impact strength increase with the increase in SiC filler content up to 10 to 15 wt.%, whereas addition of SiC content beyond 15 wt.% results in the decrease in mechanical property.
2. The value of hardness and impact strength increases up to 15 wt.% filler content; the value of tensile strength and flexural strength increases up to 10 wt.% SiC filler content, whereas an exceptional increase in the value of ILSS is noticed up to 20 wt.% SiC filler content.
3. Storage modulus values for SiC-filled glass fiber-reinforced epoxy composite decreases with the increase in SiC content. Storage modulus values decrease with the increase in the percentage of SiC content, whereas elastic range of the material increases with the increase in SiC content.
4. As the percentage of SiC content increases, the area under the curve spreads across the wider range and the peak value shifts towards the higher temperature region resulting in the increase in elastic range of the composite.
5. SiC-filled chopped glass fiber-reinforced epoxy composites represent imperfect semicircles and hence are heterogeneous in nature. Also, unfilled glass fiber-reinforced epoxy composites exhibit better interfacial characteristics and some homogeneous nature with respect to SiC-filled glass fiber-reinforced epoxy composites. The imperfect circles and heterogeneous nature is due to the uneven distribution of SiC particles inside the composite.
6. From the analysis of the results of mechanical properties and thermal properties, it has been concluded that the optimum properties are obtained for 15 wt.% SiC content in addition to 20 wt.% chopped glass fiber-reinforced epoxy composite.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

All authors read and approved the final manuscript.

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