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ORIGINAL PAPER

Low Velocity Impact, Fatigue and Visco-elastic Behaviour of Carbon/E-glass Intra-ply fibre-Reinforced Nano-silica Toughened Epoxy Composite

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Abstract
This current research dealing the effect of adding high stiffness carbon fibre along with E-glass fibre and addition of nano-silica particles as a toughening agent in epoxy resin. The main aim of this research is to improve the damping, storage modulus and fatigue behaviour of glass-epoxy composite by using carbon fibre and nano-silica particles. The intra-ply glass and carbon fibre were prepared using handloom method with uniform weft and warp. The proposed composites were prepared using hand layup method followed by post-curing at 120°C. The low-velocity impact test results revealed that the composites made with intra-ply carbon glass fibre along with 2 vol% of nano-silica offered maximum resistance against penetration. Similarly, the fatigue behaviour revealed that the presence of 1 vol% nano-silica and carbon fibre provides the highest life cycle of 30150 counts. The visco-elastic properties of glass-carbon intra-ply fibre with 2 vol% of nano-silica composite possess storage modulus of 9.5 GPa. The scanning electron microscope images revealed more shear cups in the fractured matrix, which indicates improved toughness of the epoxy matrix.

Keywords PMC · Intra-ply fibre · Nano-silica · DMA · Fatigue and damping

1 Introduction

High toughness polymer matrix composites are having huge applications in engineering starting from domestic to high-end applications [1]. Due to lightweight, the polymer matrix composites are highly preferable in automobile, aircraft and defence applications provided, if the composite posses with enough toughness [2]. In general thermoset plastics are widely used in the composite application rather than thermoplastics but the thermosets possess extreme brittleness, which is not desirable in many engineering applications [3]. These high brittle composites cannot produce high toughness and stiffness to the applied load and could failure at early loads. If the matrix is toughened using fibre or particle then the composite is more suitable for serving the damping and visco-elastic properties [4]. Generally, E-glass fibres are the one excessively used in the composite making with high toughness but still, the toughness is not improved as much as high stiffness composites in modern era. Thus these glass fibre-reinforced composites are offering low damping and fatigue properties. Many research articles have been published to point out the importance of adding high stiffness fibres in the matrix. Arun prakash et al. [5] investigated the effect of adding silicon coupling grafted iron(III) oxide particle in tensile fatigue behaviour of epoxy composite. According to the author's statement, the addition of E-glass fibre and particle into epoxy matrix improved the toughness of matrix and increasing the fatigue behaviour. Similarly, Dinesh et al. [6] studied the effect of adding bio-oil and nano-silica into epoxy resin to convert the resin as tougher one. They concluded that adding bio blender into epoxy resin increased the toughness same time reduces the thermal properties. Hence, without affecting the base properties improvement of matrix toughening is so essential and needy one. Based on the previous research, a very limited number of researchers are done their study using intra-ply model of fibre to improving the toughness. In

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this two different type of fibres may use for producing substantial outcome in composite. These intra-ply model of fibres hold good economy also when the cost factor is constrained. Moreover, the matrix toughening using nanosilica along with glass-kevlar intra-ply fibre pattern also a new way of approach. Thus based on the research gap, this current research is aimed to improve the toughness of the composite by providing a mixture of high stiffness carbon fibre along with high modulus E-glass fibre and addition of nanosilica into epoxy resin. In economical point of view the E-glass fibre is cheaper but the carbon fibre is expensive. Since the volume of fibre used for making intra-ply is almost half of the total volume, thus the cost of total product also lesser. In other hand the presence of carbon fibre offers its high stiffness along with glass fibre. Thus the composite may good with high stiffness one without increasing the product cost. The intra-ply glass/carbon fibre may prepare using handloom method since this method never requires any power and heavy-duty setup. Gokul et al. [7] reported a study on intra-ply glass-kevlar fibre preparation using handloom method. Both the yarn tex are uniform in width offering almost the same weft and warp. The proposed composites could prepare using hand layup method since it is simple and power free process. The evaluation process of prepared composites could be done following ASTM standards. These toughness improved glass-carbon epoxy composites could be used as structural member, automobile body parts, electronic components, aircraft manufacturing and defence applications [8].

2 Materials and Method

2.1 Material Used

The matrix material used for making composite is an Araldite epoxy resin (LY556) with a density of 1.18 g/cm^3 and molecular weight of 198 g/mol. The curing agent used for curing the matrix is an aliphatic hardener TETA (HY951) with a low density of 0.8 g/cm^3. The glass and carbon fibre of density 2.54 (600GSM), 1.6 g/cm^3 (280GSM) and specific gravity of 25.4, 16 were used as reinforcements and purchased from Gogreen India Pvt. Ltd, Chennai, India. The nano-silica particle of size 20 nm and density of 2.4 g/cm^3 was purchased from Sigma Aldrich, USA. All materials were used in the as-received condition without any post-treatments. Figure 1 shows TEM image of nano-silica particles used in this present study.

2.2 Making of Intra-ply Glass-carbon Woven Mat

The woven fabric form of E-glass (MIL-Y-1 140H) and carbon were prepared using handloom weaving method. In this, both the yarns of specific quantities were taken for weaving with an average roving diameter as 0.32 mm. In glass fibre is woven fabric weaving, both weft and warp consist of similar glass fibre yarns and they were weaved by the average speed of 1 square meter for every 8 h. Similar weaving trend was followed in carbon also but the time taken for weaving 1 square meter was near 9 h. This incremental in the time taken was because of high stretchability of carbon than glass fibre. Figure 2 shows the optical microscope image of warp and weft formation of glass and carbon fibre-mat made by handloom weaving method [9].

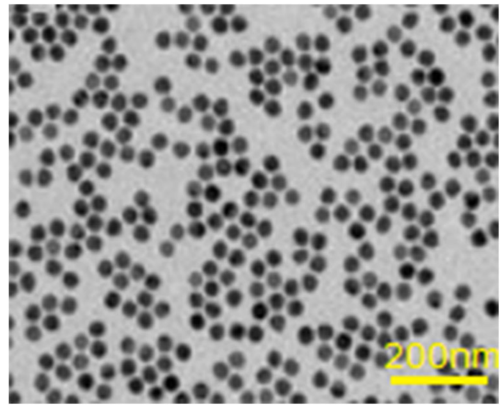

Fig. 1 TEM image of Nano-silica particles

2.3 Composite Preparation

A fixed volume of epoxy resin is mixed with the desired volume percentage of nano-silica particle and stirred continuously until the particles are thoroughly mixed. The resulted particle dispersed epoxy resin solution is then mixed with curing agent and stirred thoroughly until degassing completed. A silicon rubber mould of 3 mm thickness is used as a flexible mould to make composites. A liberal coat of liquid wax was sprayed over the silicon rubber mould before pouring the hardener mixed epoxy-particle solution for easy removal

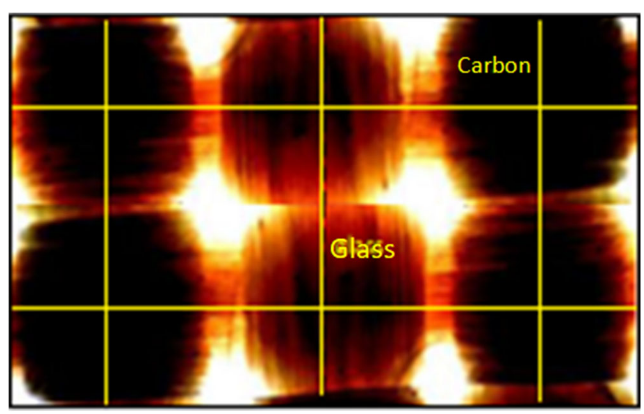

Fig. 2 glass-carbon intra ply hand loomed fibre

of cured composites. The particle mixed epoxy solution was then poured into the mould cavity and allowed to fill. 30 vol% of the glass-carbon fibre of 3 woven mats were laid one by one. Finally, the composites were cured at room temperature for 24 h and post cured at elevated temperature (120^{o}C) for 48 h. The post cured composites are then inspected for visual defects and passed to specimen making for possible testing. Table 1 shows the composition of carbon composites prepared [10].

2.4 Test Specimen Making

The visual defect inspected composite specimens were further machined for testing. The samples were machined using an abrasive water jet (Maxiem water jets, KENT, USA) with recommended process parameters. The operating pressure of 200psi, SOD of 3 mm and abrasive flow rate of 0.42 g/s are maintained as process parameters.

3 Characterization

3.1 Low Velocity Impact Damage

The low-velocity drop load impact behaviour of fabricated epoxy composites was tested using a drop load impact test machine (INSTRON-9000). The equipment had an indenter mass of 2 Kg and indenter velocity of 5 m/s. The test was conducted following ASTM D-4762 since the ply thickness is less than 0.5 mm, ply count is 3nos and specimen thickness is about 3 mm. The test samples were holed by using hydraulic clamping units for good gripping. The head of the impactor was in the form of the hemi-circular nose with 2.5 cm total length. The test was conducted a minimum five times to compute average [11].

3.2 Fatigue Testing

The fatigue behavior of composites was investigated by a tension-tension fatigue machine (MTS Landmark 370 load frame, USA) with hydraulic power actuated mechanical grippers. Dumbbell shaped specimens of five identical test samples were tested followed by ASTM D 3479 to compute average fatigue life cycles. A loading frequency of 5 Hz, stress ratio of R = 0.1, maximum load of 1.28KN (50% in maximum tensile load), the elastic modulus of 6.00 GPa, and working ambience of 25 ºC was set as process parameters.

3.3 Visco-elastic Properties

The visco-elastic properties have been evaluated based on ASTM D 4065 as temperature sweep mode. The temperature variation applied here was between 30^{o}C to 240^{o}C at a constant frequency of 1 Hz with dual form cantilever fixtures with a heating rate of 50^{o}C/min. From the test results, the storage modulus and loss tangent values were calculated.

3.4 Morphology Analysis

The fractured composite specimen’s fractograph was inspected using a scanning electron microscope (HITACH S1500, JAPAN). The samples were gold coated via sputtering for preventing of charges.

4 Results and Discussion

4.1 Drop Load Impact Test

Figure 3 shows the drop load energy absorption behavior of various epoxy composites tested. It is observed that the pure casted epoxy resin gives very lower drop load absorption of 0.35 J. This lower energy absorption is the cause of brittle nature of epoxy resin, which can’t bear the sudden load. When the sudden load is applied to the material the micro-cracks developed and further propagates and leading to plastic deformation [12]. It is observed further that the addition of E-glass fibre of 40 vol% into epoxy resin increased the drop load energy absorption. The improvement of 97.2% was observed for the composite in energy absorption. This improvement is the reason for the presence of continuous glass fibre, which could resist the sudden penetration of load. When the impactor

Table 1 Composition of carbon composites

Composite Designation	Epoxy (vol%)	Glass Fibre (vol%)	Carbon fibre (vol%)	Filler (vol%)
R	100	-	-	
RG	60	40	-	-
RGC	60	25	15	-
RGC_1	59	25	15	1
RGC_2	58	25	15	2

R-Resin; G-Glass fibre; C-Carbon.

hits the composite surface lot of momentum is transferred to the composite's surface. The conserved momentum is then distributed to fibre-reinforcement and spread over the composite. Thus the stress intensity in the impacting direction is less [13]. It is further noted that the composite (RGC), which contains carbon fibre as the addition with glass fibre improves the sudden penetration load and observed more energy. The improvement of 96.8% was observed as energy absorption for RGC composite designation. This improvement is because of the presence of high stiffness carbon fibre, which associates with glass fibre and receives a huge load and hinders the deformation. The fibre distributed load cannot move forward due to the large fibre barrier in the form of glass-carbon-glass –carbon. This nature would increase the barrier to the fast-moving impactor not to penetrate further [14].

It is observed that the presence of nano-silica along with carbon fibre further improved energy absorption. The composite designations RGC1 and RGC2 give improved energy absorption than composite made with only glass and carbon. The maximum energy absorption of 18.1 and 19.8 J, which is equal to 97.4% and 98% were observed for composites RGC1 and RGC2 respectively. This improvement is because of the presence of nano-silica fill the voids of resin and restrict the polymer molecular chains to move. The immobile polymer chains better in storage modulus and store a lot of energy [15]. Moreover, the presence of nano-silica acts like a micro crack suppresser. The nano-silica particles also provide better adhesion between fibre and particle thus the propagation of micro-cracks restricted thoroughly, which proportionally improved the energy absorption [16]. Figure 4 shows the scanning electron microscope image of the impactor penetrated (damaged) portion on composites. Figure 4(a) shows the impact damage RG composite specimen. It shows high damage portion along the impact receiving side of composite. Figure 4(b) shows the impact damage portion of glass-carbon intra-ply RGC composite designation. The fibre mangled as fine parts, which indicates a very high load barrier in the fibre pattern. Similarly, Fig. 4(c) shows the impact damage portion of RGC2 composite designation. The damage portion shows fine breakage of fibre along with matrix, which indicates effective load sharing phenomenon of fibre in nanosilica-toughened epoxy matrix. The nanosilica toughening enables the matrix to absorbs more energy while sudden load hits. Moreover, the presence of nano-silica particle also offers high adhesion with matrix, and suppresses the micro crack initiation and propagation. Thus the toughness of matrix increased further, which intern improves the impact energy absorption.[17].

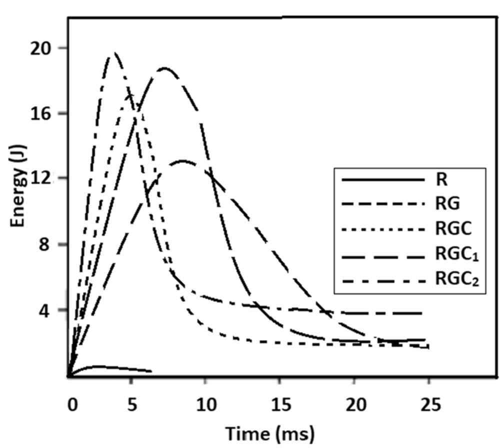

Fig. 3 Energy absorption behavior of composite

4.2 Fatigue Behavior

Figure 5 shows the fatigue behavior of epoxy and its composites. It is observed that the pure epoxy resin gives a very lower fatigue cycle of 340. This lower value is the cause of high brittle and chemically bonded molecular structure [18]. It is noted further that the addition of E-glass fibre of 40vol.% improved the fatigue life cycle as elevated. The life cycle of 22833 is observed for composite designation RG due to the effect of effective load absorption. The presence of fibre uniformly shared the load throughout the composite and maintains high dimensional stability. There no fibre-matrix interfacial cracking occurs, which indicates improved adhesion of fibre with matrix [19].

It is further observed that the addition of carbon fibre of 15 vol% along with E-glass fibre again improved the fatigue life cycle. The improved life cycle of 26142, this is equal to 13% of improvement on comparing with RG composite designation. This improvement is because of high stiffness carbon fibre, which effectively receives the load and transfers to the matrix. Thus the stress intensity used for initiating the cracks reduces and offers high retention against failure [20]. It is observed that the addition of 1 and 2 vol% of nano-silica particle further improves the fatigue life counts. The improvement of 13% and 10% were observed on comparing with RGC for composite designations RGC$_1$ and RGC$_2$ respectively. This improvement is because of the presence of nano-silica reduces the void content and improves the adhesion between particle and matrix. This adhesion improved particle-matrix interface effectively transfer the load and lowering the stress intensity factor. This lower stress intensity factor prevents the formation of interfacial cracking, which leading to improvement in fatigue strength [21]. Figure 6a and b shows the fractograph of fatigue fractured composite specimens. The Composite RGC$_1$ (Fig. 6a) shows improved adhesion of fibres without interfacial cracking than RGC composite (Fig. 6b) designation, which posses with interfacial delamination.

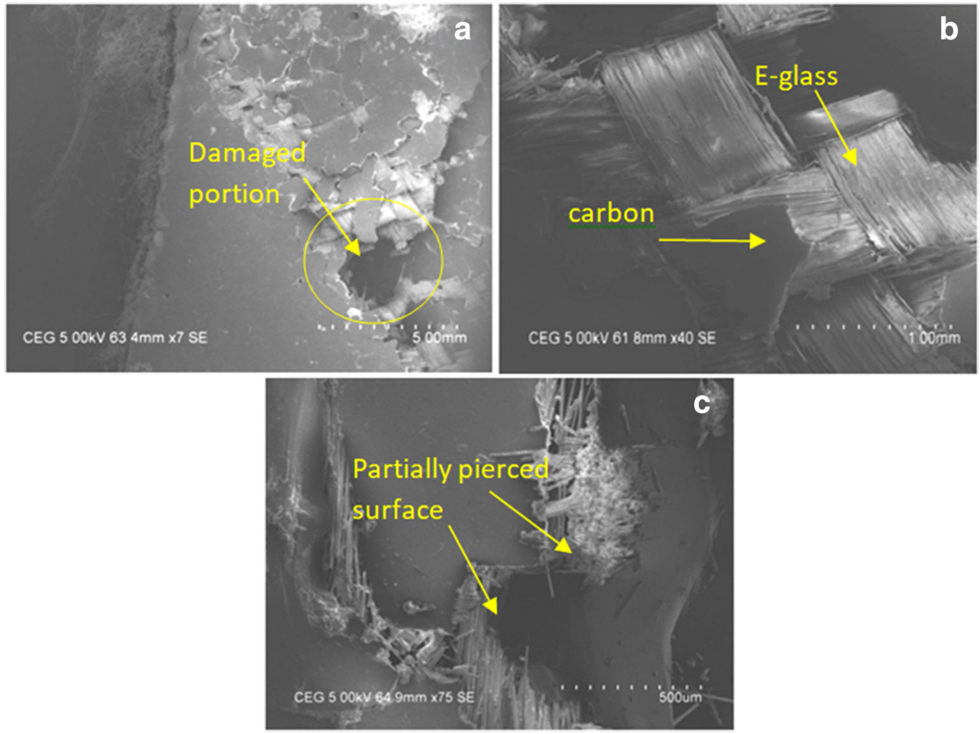

Fig. 4 SEM images of (**a**) EG, (**b**) EGC and (**c**) EGC_2 composite designation

4.3 Visco-elastic behaviour

Figures 7 and 8 shows the visco-elastic properties of epoxy and its composites. Figure 7 shows the storage modulus and 8 shows the loss tangent factor. It is observed that the pure epoxy resin gives very lower storage modulus of 1.8GPa at a lower temperature. When temperature increases beyond its glass transition the storage modulus goes even lesser. This is because of no energy-absorbing mechanism presence within the neat resin. When temperature increases the secondary molecules, which are attached with primary C-C chain starts rotating and increases in free volume thus reduce the storage modulus [22]. It is further noted that the addition of glass and carbon fibre increases storage modulus further. The composite designation RG and RGC give improved storage modulus of 4.6 and 5.6GPa respectively. This improvement is the reason for the presence of high heat capacity glass fibre and thermal conductivity carbon fibre, which offers very high energy absorption in lower temperature and even higher temperatures [23]. Similarly, the addition of nano-silica of 1.0 and 2.0 vol% further improves the storage modulus. The composite designations RGC_1 and RGC_2 give storage modulus up to 8 and 9.4GPa respectively. This huge improvement is because of two reasons. Firstly the presence of nano-silica particle absorbs the high frequency repeated load, temperature and retains the stability. The presence of silica particle receives the load and distribute the same uniformly in the composite. Thus the stress concentration in the matrix medium reduces and stable for larger repeating cycle. Similarly, the high specific heat of nano-silica up to 680 J/kg.K absorbs the heat energy, which is given to the composite and hinders the rotation of secondary molecules in primary epoxy C-C molecular chain. Thus more heat energy is required to activate the secondary molecules, which intern improves the dynamic mechanical stability. Secondly, the nano-silica particles filled in the polymer molecular voids and reduce the mobility of the molecular chain. Thus a lot of energy may require activating the secondary molecules of epoxy resin, thus higher storage modulus is observed [24].

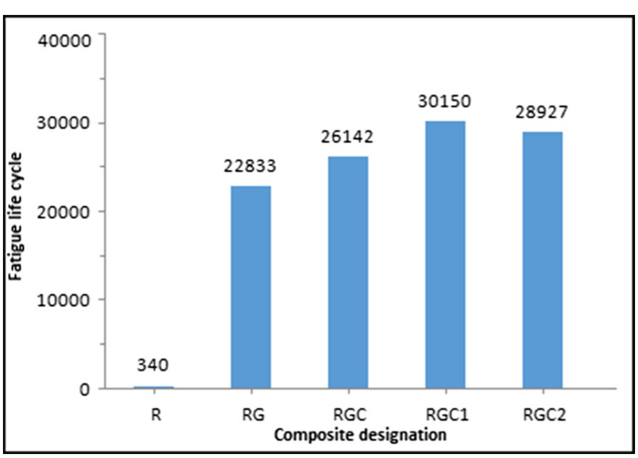

Fig. 5 Fatigue behavior of composites

Figure 8 shows the loss factor of epoxy and its composites. It is observed that the pure epoxy resin gives a higher loss factor since it could not bear the load and increase of

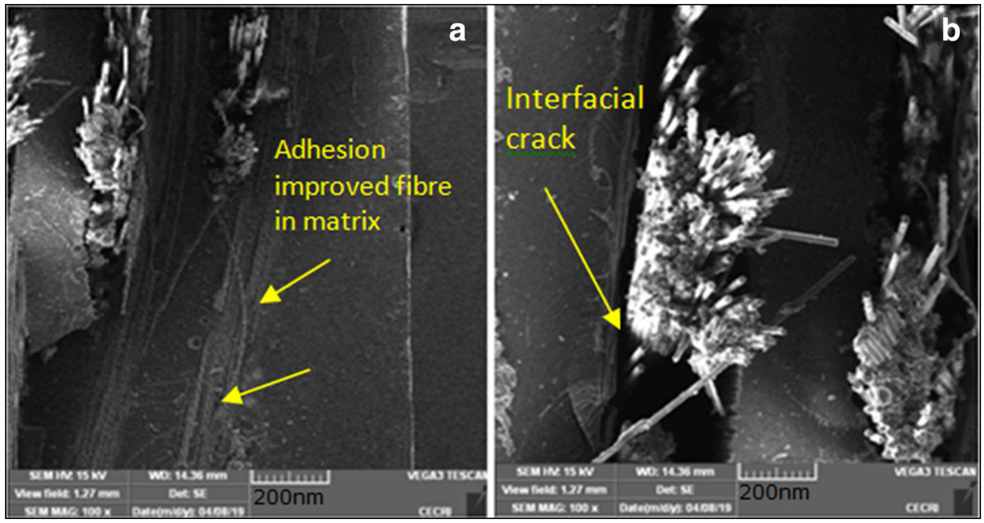

Fig. 6 SEM images of fatigue fracture specimen (a) RGC_1 and (b) RGC

temperature. When the temperature is equal to glass transition temperature the loss factor is very high. This phenomenon is because of at glass transition temperature the epoxy secondary molecules start rotating and not retain any plastic strain, thus high loss factor is observed. It is observed that further additions of glass and carbon fibre of 40 vol% marginally reduces the loss factor. A lower loss factor of 0.95 is observed for composite designation RGC at 65°C. It is noted that the glass transition is slightly shifted towards higher value due to the presence of fibre element [25]. The addition of 1 and 2 vol% of nano-silica further reduces the loss factor. A very lowest loss factor of 0.8 at 84°C is observed for composite designation RGC_2 at 1 KHz. This lowest loss factor is the reason for high plastic strain retention of the nano-silica particle in the epoxy matrix. The presence of silica particles keeps absorbing the strain, which is given through the heating and loading process and reduces the easy mobility of polymer chains, thus showing very lowest loss factor [26, 27].

5 Conclusion

The carbon-glass intra-ply fibre-reinforced epoxy resin composite was prepared using nano-silica particles for improving the visco-elastic, fatigue and drop load impact behavior. The intra-ply glass and carbon fibre were prepared using the hand-looming method and the proposed hybrid composites were prepared using hand layup method.

- The low-velocity drop load impact test revealed that the addition of high stiffer carbon fibre along with glass fibre improved the penetration resistance. Further addition of 2 vol% of nano-silica particle further improved the energy absorption.
- The fatigue behavior resulted in good improvement in 1 vol% of nano-silica dispersed glass-carbon epoxy composite. The addition of nano-silica particles improved the adhesion between fibre and matrix, thus interfacial debonding suppressed.

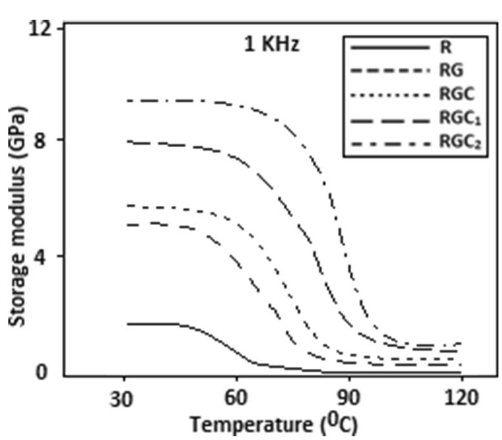

Fig. 7 Storage modulus of composites

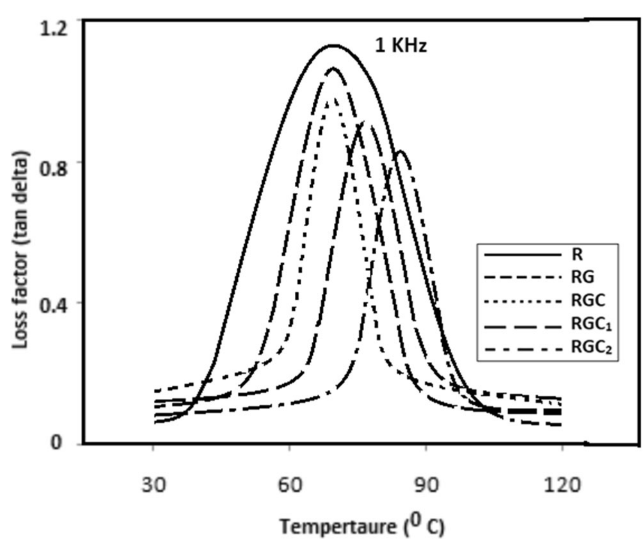

Fig. 8 Loss tangent of composites

- The visco-elastic behavior of nano-silica dispersed glass-carbon intra-ply epoxy composite shows improved storage modulus and lower loss factor. The nano-silica volume of 2% fetches higher storage modulus and loss factor.
- Thus the addition of carbon fibre along with glass fibre improves the properties like low-velocity drop penetration, fatigue and visco-elastic properties, whereas addition of nano-silica particles further improves the above-mentioned properties.
- These fatigue strength improved, high load penetration resistance and high storage modulus with low loss factor epoxy composite could be used in many engineering applications based on the consumer need.

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