Enhancement in Mechanical and Thermal Properties of Glass/Epoxy Composites Through Incorporation of Nano-Al₂O₃ Particles

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Abstract—Fiber reinforced polymeric (FRP) composite materials are at present discovers abundant structural and materials associated applications. These composites exhibited damage and degradation at the time the in-service time due to different environmental parameters. To restrict degradation from various environmental factors and enhance the mechanical and thermal behavior of the glass/epoxy (GE) composites; nanofillers are added in the polymer matrix. The current research is intended to evaluate the effect of addition of nano-Al₂O₃ particles on the mechanical and thermal behavior of GE polymeric composites. In order to estimate the improvement in the mechanical properties, tensile tests of the modified samples were carried out at 1 mm/min loading rate. The epoxy matrix was altered with various nano-Al₂O₃ content (i.e. 0.1, 0.3 and 0.5 wt. %). The tensile strength of 0.1 wt.% nano-Al₂O₃/GE filled composites exhibited higher ultimate tensile strength (UTS) among all other composites. Similarly, in case of flexural testing; flexural strength of 0.1 wt.% nano-Al₂O₃/GE filled composites revealed the maximum strength value. The possible reason may be attributed to the good dispersion of nanoparticles in polymer matrix corresponds to proper stress transfer during addition of 0.1 wt.% nano-Al₂O₃ particles. In order to access the variations in the viscoelastic behavior and glass transition temperature due to the addition of nano-Al₂O₃ fillers in GE composites, dynamic mechanical thermal analysis (DMTA) measurements were carried out. Different types of failures and strengthening morphology in the composites were analyzed under scanning electron microscope (SEM).

Keywords—Glass fiber/epoxy composite; Nano-Al₂O₃; Ultimate tensile strength (UTS); Flexural strength; Dynamic mechanical thermal analysis (DMTA)

I. INTRODUCTION

The usage of composite materials over the conventional metallic materials is nowadays increasing in rapid rate as compared other materials. In composites, especially the use of fiber reinforced polymeric (FRP) composites materials in structural as well as in daily usable components are being in practice. The reason for vast usage of these FRP based materials lies in its enhanced mechanical and thermal properties. These composites are globally being used in many fields such as structural, sporting goods, aerospace, automotive, low temperature applications, gas and oil pipelines. But in real time applications these materials are subjected to various harsh and hostile environmental parameters [1]. In results damage and degradation occurred to the FRP composites materials. Therefore, to minimize these environmental damage and degradation and to enhance the various mechanical and thermal properties of the composites; different nanofillers are being incorporated into the composites through matrix modification [2–4]. The addition of nano-Al₂O₃ particles improves bearing strength [5], storage modulus [6] and glass transition temperature [7], flexural properties, thermal conductivity [7].

In the present experimental study, epoxy matrix was modified with different nano-Al₂O₃ content and the required FRP laminates was prepared. The effect of incorporation of nano-Al₂O₃ particles on the mechanical and thermal behavior of GE polymeric composites was investigated. In order to estimate the improvement in the mechanical properties, tensile tests of the modified samples were carried out at 1 mm/min loading rate. The thermal properties of the FRP composites were carried out using dynamic mechanical thermal analysis (DMTA) measurements.

II. EXPERIMENTAL TECHNIQUE

A. Materials

The epoxy matrix used in the investigation was well standardized with the trade name as diglycidyl ether of bisphenol A (DGEBA) and hardener having the vendor name as triethylene tetra amine (TETA). The reinforcement used in the investment was woven fabric E-glass fiber. The orientation of fibers in warp and weft direction was 0⁰ and 90⁰ respectively. The glass fiber was obtained from Owens Corning Industries. The resin (epoxy) and hardener are picked up from Atul Industries, Gujarat defining the manufacturer trade name as Lapox L-12 and K-6 respectively. During fabrication of glass/epoxy composites, the resin to hardener weight fraction was taken as 10:1. The mechanical properties of epoxy and glass fibers are shown in table I.

<table>
<thead>
<tr>
<th>TABLE I: MECHANICAL PROPERTIES OF EPOXY AND GLASS FIBER</th>
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<tr>
<td>Properties</td>
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<tr>
<td>Density (g/cm³)</td>
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<td>Tensile modulus (GPa)</td>
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<td>Tensile Strain (%)</td>
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<td>Areal weight of fabric (g/m²)</td>
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B. Preparation of control GFRP and nano-Al₂O₃ enhanced GFRP composites

Epoxy and glass fiber was taken in 40:60 weight ratios. Required amount of nano-Al₂O₃ particles were mixed with acetone and stirred by a magnetic stirrer for 30 min. Then sonication of the nano-Al₂O₃ particles and acetone was done for 30 min. In the meantime epoxy matrix was heated at 120 °C temperature. Then the epoxy matrix, nano-Al₂O₃ particles and acetone was mixed in a beaker and magnetic stirring was done till the complete evaporation of acetone from the solution. Vacuum degassing of the solution was done for 18 h in a vacuum chamber to removes the bubble formation at the time of magnetic stirring and sonication. The hardener (10 wt. % by weight of epoxy matrix) was mixed with the epoxy very smoothly to avoid any formation of bubbles. The FRP composite laminates were fabricated by hand lay-up method. Using hot press hydraulic machine the laminates were pressed at 60 °C for 20 min. The cured laminates were cut using a diamond coated cutter into different dimensions as per the requirement testing such as tensile and dynamic mechanical thermal analysis evaluation. Furthermore, to ensure proper curing of composites specimens are cured at 140 °C for 6 h [8].

C. Different test parameters for control GE and nano-Al₂O₃ enhanced composite

In order to ensure the durability and reliability of nano-Al₂O₃ composites are very important for different engineering applications. A Universal Testing Machine (UTM) of INSTRON 8862 was used to evaluate the tensile strength as per ASTM D3039 standard. The dimension of the sample was 250 mm X 25 mm X 2.5 mm (LxWxT). Loading rate of 1 mm/min was maintained throughout the test. There are minimum of five specimens of each type has been considered for the test and the average values with standard deviation are reported. DMTA measurements were carried out to assess the viscoelastic properties and glass transition (Tg) performance of the composites. Scanning electron micrography (SEM) was carried out for the fractured specimens to define the strengthening mechanisms and accumulation of nano-Al₂O₃ particles.

III. RESULTS AND DISCUSSION

A. Effect of tensile behavior of nano-Al₂O₃ enhanced GE composites

The stress vs strain graph for control GE, 0.1, 0.3 and 0.5 wt. % nano-Al₂O₃ enhanced GFRP composites tested at a loading rate of 1 mm/min. It was evident from fig. 1 that the tensile strength of 0.1 wt. % GE/nano-Al₂O₃ composites revealed higher strength among all other composites. Further, addition of nano-particles into the epoxy matrix shows decrease in the value of overall strength of the composites. The decrease in strength may be attributed to accumulation of nano-Al₂O₃ particles at a particular area in the epoxy matrix. This bunches of nano-Al₂O₃ particles creates poor transfer of load along the direction of fiber/matrix interfacial area.

Fig. 1. Stress Vs. Strain curve for control GE and nano-Al₂O₃ enhanced GE composites at several nano-Al₂O₃ particles contents

Fig. 2 indicates the variation in tensile strength and modulus acquired up from fig.1. Fig. 2 was plotted against different nano-Al₂O₃ content for unmodified GE and nano-Al₂O₃ enhanced GE composites. By addition of 0.1 wt % nano-Al₂O₃ in the GE composite, improvement in UTS as well as in modulus value than control GE composites as shown from fig. 2(a) and fig. 2(b). By the incorporation of 0.1 wt.% nano-Al₂O₃ particles improved the tensile strength by 15.32% than control GE composite. This enhancement in strength could be governed by the high specific surface area of nano-Al₂O₃ particles that possesses high interfacial zone across the composite. However, for proper stress transfer this high specific surface area makes easy across the interfacial area. But, with further accumulation of nano-Al₂O₃ particles (0.3 and 0.5 wt. %) in the GE composites owed to reduction in modulus and strength values. This reduction in strength and modulus corresponds to the accretion of nano-particles in the epoxy matrix of the composite at a specific region.

Fig. 2. Difference in (a) Tensile strength and (b) modulus with GE and nano-Al₂O₃ filled GE composites

Typically, at the interfacial region poor wettability causes to weaker adhesion between the nano-Al₂O₃ particles, fiber and matrix and this causing accretion of nano-Al₂O₃ particles. The tensile strength of nano-Al₂O₃ filled GE composite with 0.1 wt. % nano-Al₂O₃ was found to remain higher as compared to rest other composites system. The modulus vs nano-Al₂O₃ content of GE composites was revealed in fig.2(b). The modulus was found to be 5.27 % higher in case of 0.1 wt. % nano-filler enhanced GE composite as related to control GE. This improvement in modulus value corresponds to proper bonding behavior of matrix/fiber/ nano-Al₂O₃ particles.
particles in composite. Fig. 3 reveals the failed images of GE and nano-Al₂O₃ enhanced GE composite with several failure patterns tested after the tensile tests at a 1 mm/min loading rate. The sample A reveals fracture at the middle part of the composite, while sample B, C and D indicates cracks mostly near the tab area of the composite samples.

![Failed configurations of (a) Unmodified GFRP (b) 0.1 % nano-Al₂O₃, (c) 0.3% nano-Al₂O₃ and (d) 0.5 % nano-Al₂O₃.](image)

**B. Dynamic Mechanical Thermal Analysis (DMTA) of thermal shocked conditioned composites**

The thermo-mechanical investigation of control GE and nano-filler enhanced GE composites was done in the DMTA (Model: Netzsch DMA 242E) in between 30 °C to 200 °C temperature. The specimens were prepared ASTM D7028 standard. The heating rate used during experiment was 5 °C/min. The mode of fixture used in DMTA was 3-point bending. During testing of samples 5 Hz frequency was maintained. The apparatus supplies dynamic stress to the sample and the outcome was examined in terms of dynamic displacement. The supplied stress and produced strain keep on in a phase in case of a perfectly elastic material. However, there was a phase variation seen in case of polymeric (i.e. viscoelastic) composite material. The elastic modulus of the material was represented in terms of storage modulus (E′). Whereas, the viscous modulus represents the loss modulus (E″). The coefficient of damping (tan δ) was determined between the fractions of E″ to E′. Fig. 4(a)-(d) represent the deviation in E′, E″, tan δ and glass transition behavior (Tg), with temperature alteration for control GE and nano-Al₂O₃ filled GFRP composites. Fig. 4(a) depicts that the E′ for 0.1 wt.% nano-Al₂O₃ enhanced GE composite was higher as that of control GE composite at temperature below Tg. The inception of sharp alteration in slope of E′ with temperature was measured as the Tg of the corresponding composites. Fig. 4(b) shows the variation in E″ owing to fusion nano particles with the GE composites.

The value of E″ was measured maximum at 0.1 wt.% nano-filler enhanced GE composite. The tan δ value reduces as the nano-Al₂O₃ particles starts to accumulate presenting higher brittle behavior in the composite as observed from fig. 4(c). Fig. 4(d) shows that fusion with 0.5 wt. % nano-Al2O3 particles in the GE composites reduces the Tg from 115.3 °C to 102.6 °C. As the content of nanofillers increases in the GE composites the Tg values goes on decreasing. The decrease in Tg value may be attributed to the interference of progresses of crosslinks due to entrapment of nano-Al₂O₃ particles in the polymer chains. This decrease in Tg temperature may be attributed to the rearrangement of the polymeric chains in the interfacial area.

**C. Fractography analysis by SEM**

SEM was carried out for nano-Al₂O₃ filled GE composites and different strengthening morphology and reduction in strength value of composites were studied and are shown in fig. 5. Fig. 5(a) illustrates 0.1 wt. % nano-filler filled GE composite showing good scattering of nano-Al₂O₃ particles all over the epoxy matrix. Appropriate scattering of nano-filler throughout the PMC carries proper stress transfer across the epoxy matrix to the fiber. Lump of nano-Al₂O₃ particles at a particular area in epoxy matrix are seen in case of 0.5 wt.% nano-filler enhanced GE composites as shown in fig. 5(b). This aggregation of nano-Al₂O₃ particles at a particular zone of epoxy matrix corresponds to inappropriate stress transfer through the matrix phase following with decrease in modulus and strength values. In case of 0.1 wt. % nano-Al₂O₃ bridging phenomenon was observed between the nanoparticles and epoxy matrix region as shown in fig. 5(c) and 5(d).
IV. CONCLUSIONS

The present investigation of control GE and nano-Al2O3 filled GFRP composites have the resulting pertinent outcomes as follows;

- The tensile strength of 0.1 wt.% nano-Al2O3/GE filled composites exhibited higher UTS compared to all other composites.
- With the incorporation of 0.1 wt.% nano-Al2O3 in the GE composite improved the tensile strength and modulus by 16.22% and 5.27% respectively than control GE composite.
- DMTA analysis reveal that the storage modulus was found to be maximum for 0.1 wt.% nano-Al2O3/GE filled composites and Tg decreases with increase in nano-Al2O3 content.
- SEM micrographs shows strengthening mechanisms by crack bridging phenomenon between nanoparticles in 0.1 wt.% nano-Al2O3 filled GE composites, whereas in higher percentage enhanced GE composites indicates agglomerations of nanoparticles.

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