Comparative Analysis of Flexural Strength of Silicon Dioxide Epoxy Nanocomposite and Graphene Epoxy Nanocomposite

Kriti Sahai and Audhesh Narayan
Department of Mechanical Engineering, Motilal Nehru National Institute of Technology, Allahabad, Prayagraj, Uttar Pradesh, India

Abstract
Polymer nanocomposites (PNCs) have recently grown in material research because of their dominant applications and strength compared to their counterparts. In this study, Silicon Dioxide (SiO\(_2\))-Epoxy Nanocomposite (SENC) with 1%, 2%, and 3% nano-silica fillers by weight reinforced in epoxy and 0.5% weight of Graphene in Epoxy Nanocomposite (GENC) is made with solution casting method. The specimens were tested for flexural strength. The comparative plots of stress-strain and force-extension are drawn for neat epoxy along with 1% SENC, 2% SENC, and 0.5% GENC. It is observed that 3% SENC shows better flexural strength than neat epoxy, 1% SENC, 2% SENC, and 0.5% GENC. Also, GENC shows better toughness and stability even at a low fractional concentration of 0.5% compared to neat epoxy, 1%, and 2% SENC. However, 0.5% GENC has lower flexural strength than 3% SENC.

Keywords
Polymer nanocomposites, Silicon dioxide epoxy nanocomposite, Graphene epoxy nanocomposite, Flexural strength, Nanoparticle concentration

Introduction
The PNCs may be considered matrices loaded with reinforcement or nanofiller. It is a mixture of two or more materials homogeneously dispersed in a polymer matrix, where at least one dimension of the reinforcing agent is smaller than 100 nm. Manufacturers select a suitable concentration of nanoparticles loading in a polymer matrix to enhance mechanical, thermal, and other properties without affecting their processing method. A harmonious combination of nanofillers and epoxy matrix in nanocomposite fabrication reduces manufacturing costs and improves other properties, such as thermo-mechanical, viscoelastic, or explosive properties. Sometimes adding more than the required weight concentration of particulate fillers contributes drawbacks to the resulting composites, such as opacity, brittleness, or lack of desirable properties.

The reinforcing effect of nanofiller depends on several factors, such as properties of the particle, shape and size, the nature of nanoparticles and their concentration, type of nanofiller in a polymer matrix, nanoparticles aspect ratio, nanoparticles orientation, and nanoparticles distribution. Various types of nanoparticles, such as Alumina, Zirconia, Titanium Dioxide, SiO\(_2\), Carbon Nanotube, Graphene, and many more, have been used to obtain nanocomposites with various polymers-based matrix such as Epoxy, Natural rubber, etc., based on their applications.

PNCs exhibit unique physicochemical properties that cannot be obtained with individual components acting alone. Recent research and findings in the field of PNCs show the importance and usefulness of PNCs for everyday real-life
applications. SENC is used popularly in photovoltaic cells, Light Emitting Diodes and photodiodes, printable conductors, optical displays, many catalytic, the base for electrical devices, mechanics, photoconductors, and superconductor devices. GENC is used in electrocatalysis, batteries, and chemical sensors for detecting and removing heavy metal ions, organic pollutants, solar energy conversion and electrochemical-electroluminescent devices, bright windows, and memory devices [1]. The properties of these nanocomposites are still a hot topic of research, giving way to more diversified applications. The properties of reinforced polymer matrix strongly depend on the nanometric inorganic compounds in polymers so that the properties are improved.

Tyagi et al. [2] discussed carbon nanotubes as fillers and their application in the electronics industry. Alexandre et al. [3] reported current expansion in synthesis, properties, and future applications of polymer-layered silicate nanocomposites. LeBaron et al. [4] defined three methods of fabrication of PNCs in which the in-situ polymerization method is used for clay-based nanocomposite. In contrast, in-situ and melt intercalation techniques effectively produce reinforced polyethylene hybrids. It is also reported that, in general, composite films were obtained by the Solvent Casting technique. Young et al. [5] reviewed the intrinsic mechanical properties of the graphene family. They concluded that for nanocomposites, it is incorrect to assume that the filler modulus does not depend on the matrix, thereby explaining the performance of graphene as filler in some matrix systems.

Mohan et al. [6] performed a Taguchi analysis to find the optimal blend of graphene derivatives with thermoplastic polymer matrices and found improved mechanical properties when 3 wt.% and 6 wt.% graphene’s are loaded in polypropylene and polyethylene matrix and showed GENC loaded with 0.52% nanofiller by volume made with solution casting have 2.54 x 10⁶ S/cm electrical conductivity.

Sprenger [7] conducted an exhaustive literature survey and concluded that nano-silica particles are monodisperse, and no agglomerations occur even at very high concentrations. Rafiee et al. [8] discussed that graphene content with a lower fractional value near -0.1 wt.% in epoxy systems as a requirement of graphene in smaller fractional weight, together with the inherently lower production costs adds an advantage to processing GENC as compared to other competing nanofillers.

Singh et al. [9] explored the properties of SENC and reported that by raising the nano-silica dispersion from 1 to 4% by weight, improvement in all properties of nanocomposites is observed. It is also concluded that the mechanical properties of nanocomposites deteriorate by adding nano-silica above 4% by weight in the epoxy matrix. Singh et al. [10] studied the effect of post-curing temperatures on mechanical and viscoelastic properties of SENC when spherical nano-silica particle (15 nm diameter) concentration in the epoxy matrix rises from 2 to 8% by weight. Rajpoot et al. [11] successfully dispersed 2% to 8% SiO₂ nanoparticles in epoxy resin using ultrasonication and found a significant improvement in fracture toughness and fracture energy of SENC with four wt.% nanoparticle loading in the epoxy matrix, after which there is deterioration in properties of SENC. Different nano-silica fillers concentration is used in this study to fabricate SENC, and 0.5 wt.% of graphene nanofillers are selected for GENC in this study for comparative flexural analysis.

**Materials and Methods**

**Selection of material**

S-type nano-silica and graphene nanoparticles are purchased and first analyzed using Particle Size Analyzer (PSA; Model: Microtrac, Nanotrac Wave II/Q Zeta) at CIR Laboratory MNNIT Allahabad. PSA determines the particle size and distribution in liquid dispersions using the backscattered laser amplified reference method, where the laser is passed through the particles dispersed in the fluid medium. The smaller particles scatter light at larger angles than a larger particle that scatters light at smaller angles. This angular deviation is measured in light intensity scattered as the laser beam passes through the particulate dispersion. The results analyzed using PSA show 99.9% pure white spherical nano-silica fillers as 30 - 50 nm in size (Figure 1a) and graphene nanoparticles as 15 - 30 nm size (Table 1 and figure 1b). Figure 2 shows XRD (X-ray diffraction) analysis of graphene nanoparticles. Epoxy resin LY556 and Hardener HY IN 951 mixed in a 13.5:1 ratio is used for nanocomposite fabrication.

**Method of nanocomposite preparation**

Solution mixing is a novel and most commonly used approach for SENC and GENC fabrication. A composite without any particle concentration termed neat epoxy is first fabricated to compare the strength with the epoxy matrix loaded with 1, 2, and 3 percent nano-silica and 0.5 percent graphene nanoparticles by weight as a reinforcing agent.

SiO₂ nanoparticles are first measured using a weighing balance (Model: Shimadzu ATX 224) for 1, 2, and 3 percent by weight of particles concentration, respectively. SiO₂...
nanoparticle are then kept in a hot air oven for 1.5 hours at 50 °C to remove moisture. The nanoparticle’s weight is measured once again before mixing it with 50 gm ethanol solution and then ultrasonicated for 1 hour at 10 Hz. Ethanol is used for uniform dispersion of SiO₂ nanoparticles in solution. Also, it lowers the viscosity of the epoxy solution. After ultrasonication, it is stirred with a glass rod and mixed with preheated LY556 epoxy solution. The mixed solution is stirred on a hot plate magnetic stirrer at 70 °C for 2 hours. Ethanol is removed from the mixture by heating at it 70 °C. The removal of ethanol was ensured by measuring the weight of the mix before and after removal. The solution is kept in a degasser to remove the air bubbles trapped in the mixture.

Hardener is mixed with the solution in a 13.5:1 ratio, stirred for 2 minutes, and poured into molds of size 8.5 mm x 4 mm x 4 mm x 4 mm. Then it is left at room temperature for 24 hours and removed from the mold once cured. This same approach has been used for fabricating 1, 2, and 3 percent by weight of SENC (Figure 3) and 0.5 percent by weight of GENC.

Three-point flexural test

A 3-point Flexural Test is performed on a computer-controlled Universal Testing Machine Tinius Olsen 10 kN (Figure 4). The 3-point Flexural Test measures the bending force required for the beam of composites, plastics, or similar material and determines the flexing resistance or stiffness of a material. The flexural modulus of the material is determined using the slope of stress vs strain deflection curve.

Results and Discussion

A load of 10 kN on the span of 40 mm with a moving speed of 1 mm/min was used to perform 3 points bending test with five samples tested for neat epoxy, SENC, and GENC each. The dimension of SENC is 85 mm x 40 mm x 4 mm, and GENC is 77mm x 40 mm x 3.5 mm. Figure 5 shows the force vs extension plot for Neat Epoxy, 1, 2, and 3 percent by weight SENC compared to 0.5 percent by weight GENC. It was found from the force-extension curve (Figure 5) that Neat Epoxy has a flexural strength of 27.11 MPa. Also, for neat epoxy, a 13.57 MPa strength is generated at 1% stress with maximum strain and strain at break point equal to 1.719%.

1% nano-silica in the epoxy matrix specimen are tested and have a flexural strength of 28.71 MPa. The stress of 9.11 MPa in specimen is located at 1% of the test sample’s original height having maximum strain as 2.750% and break strain equal to 2.750%. For 2% SENC, flexural strength is 69.0 MPa, with stress at 1% of the test sample’s original height equal 18.56 MPa. 2% SENC has the maximum strain and breakpoint strain of 1.719%.

From the above force-extension plot for 3% SENC in figure 5, it can be observed that there is an overall improvement in flexural strength of 50.3 MPa, having the stress of 8.77 MPa and 36.96 MPa for 1% and 3.5% of the test sample’s original height, respectively. The maximum strain developed is 5.76%, and the strain of 9.49% at the point of the break. The plot shows that 3% nano-silica fillers addition in the matrix enhances the potential load/stress bearing capabilities of SENC, thus providing better mechanical characteristics than its counterparts.
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The comparative force-extension plot for 0.5% GENC, in figure 5, depicts flexural strength of 37.67 MPa and 20.48 MPa stress at 1%. With this, GENC has maximum strain % and strain at a breakpoint of 1.732%. A stress-strain deflection curve is plotted with flexural test force extension values for SENC and GENC. The stress-strain deflection graph in figure 6 for SENC and GENC shows that 3% SENC has more significant elongation when subjected to load compared to neat epoxy, 1% and 2% and even better than 0.5% GENC, thus showing better strength and toughness.

Also, flexural strength shows an improvement of 85.54% as nano-silica concentration increases from 1 to 3 percent by weight concentration. The flexural strength of SENC after 4% decreases due to particle aggregation and clustering.

Even at a low fractional percentage of 0.5% graphene nanoparticles reinforced in epoxy matrix, GENC shows outstanding flexural strength and toughness. Thus, graphene can be a low-cost substitute for potential applications.

Conclusions

Literature has reported an improvement in the thermo-mechanical properties of SENC by adding nano-silica as fillers in epoxy matrix up to 4%, after which the properties deteriorate due to particle agglomeration or clustering.

- The strength of PNCs increases with the addition of nanofillers.
- For 1% and 2% by weight in SENC, there is an improvement of 58.8% in the strain at a breakpoint and an 81.8% rise in breakpoint strain for 3% SENC.
- 3% SENC shows approximately 80% more flexural strength as compared to neat epoxy, 1% and 2% SENC and 0.5% GENC. It can bear the highest stress of 36.96 MPa at 3.5% of the test sample’s original height.
- The curve of the stress-strain plot depicts the improvement in mechanical strength of SENC by increasing the filler concentration up to 3%.
- From the stress-strain deflection curve, it is observed that graphene, even at a very low fractional of dispersion in an epoxy matrix, shows better flexural strength and toughness as compared to nano-silica dispersed in an epoxy medium.

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Conflict of Interest

The authors do not have any conflict of interests to reveal.

Credit Author Statement

Kriti Sahai: Experimentation, Data curation, Writing - original draft preparation, Writing - review and editing; Audhesh Narayan: Experimentation, Writing - original draft preparation, Writing - review and editing, Supervision. All the authors read and approved the manuscript.

References


Table 2: Summary of various concentrations of nanoparticles mixed with epoxy resin and hardener.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Epoxy wt. LY556</th>
<th>Hardener ARDUR HY 951 IN</th>
<th>Particle wt.</th>
<th>Flexural Strength</th>
<th>Stress at 1%</th>
<th>Stress at 3.5%</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(g)</td>
<td>(g)</td>
<td>(g)</td>
<td>(MPa)</td>
<td>(MPa)</td>
<td>(MPa)</td>
</tr>
<tr>
<td>Neat Epoxy</td>
<td>100</td>
<td>13.5</td>
<td>0</td>
<td>27.11</td>
<td>13.57</td>
<td>-</td>
</tr>
<tr>
<td>SiO₂ 1% Np Conc.</td>
<td>100</td>
<td>13.5</td>
<td>1</td>
<td>28.71</td>
<td>9.11</td>
<td>-</td>
</tr>
<tr>
<td>SiO₂ 2% Np Conc.</td>
<td>100</td>
<td>13.5</td>
<td>2</td>
<td>69.0</td>
<td>18.56</td>
<td>-</td>
</tr>
<tr>
<td>SiO₂ 3% Np Conc.</td>
<td>100</td>
<td>13.5</td>
<td>3</td>
<td>50.3</td>
<td>8.77</td>
<td>36.96</td>
</tr>
<tr>
<td>G 0.5% Np Conc.</td>
<td>100</td>
<td>13.5</td>
<td>0.5</td>
<td>37.67</td>
<td>20.48</td>
<td>-</td>
</tr>
</tbody>
</table>

Figure 6: The stress-strain deflection graph for SENC and GENC.
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