Visual Abstract

# Mechanical and Fracture Characterization of Epoxy/PLA/Graphene/$SiO_2$ Composites

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**Abstract.** This work investigated the effects of graphene and $SiO_2$ addition on the mechanical and fracture properties of epoxy (80%) – Poly Lactic acid (PLA) (20 %) composites. Epoxy-PLA composites were loaded with graphene and $SiO_2$ (0.1-0.5 wt. % with equal weightage of each filler), and were manufactured by bath sonication followed by manual casting. The tensile, flexural strength and fracture toughness of nanocomposites increased with an increase in filler concentration till it reached 0.3 wt. %. The addition of filler content higher than 0.3 wt. % drastically reduced the mechanical and fracture properties. The fractured surfaces from the tensile tests were examined using Scanning electron microscopy (SEM) imaging to understand the effects of filler addition. The numerical analysis was also performed to simulate the impact of filler concentration on the tensile strength of nanocomposites using representative volume element (RVE) in ANSYS workbench.

**Keywords.** Graphene, $SiO_2$, Mechanical, Fracture testing, SEM.

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## Introduction

Because of their wide range of desirable properties, synthetic and biopolymer-based materials play a vital role in daily life. When compared to traditional materials, these polymers have a high strength-to-weight ratio [1-2]. Because of the increasing scarcity of petroleum resources and the resulting pollution, petroleum-based polymers must be replaced with environmentally friendly and biodegradable materials [3].

As opposed to its equivalent micro-based materials, nanotechnology, and nanomaterials have incredibly desired features, which is why they are becoming more and more popular. The dispersion of fillers in the holding matrix is still a difficult process for the infusion of greater dosages of nanoparticles (NPs) because it causes agglomeration. Nanocomposites are materials that have been the subject of extensive investigation. Researchers are investigating workable options to guarantee the consistent distribution of nanofillers in the holding matrix using various methods. Due to their superior consistency, and structural, electrical, and mechanical properties, polymer nanocomposites are demonstrating significant potential as a future material in a variety of technical applications [4].

Epoxy is a thermosetting polymer with varied qualities including mechanical, thermal, low density, and high brittleness. It can be used as a matrix, adhesive, and coating. Many researchers work to make the epoxy-based material less brittle to increase its ductility. Epoxy-based nanocomposites have found applications in the automotive, aerospace, sporting goods, marine, and biomedical industries.

Graphene and multi-walled carbon nanotubes (MWCNTs) are used to create lightweight nanocomposites that have improved mechanical, electrical, thermal, and optical properties. In comparison to non-functionalized graphene material, higher dosages of graphene nanoplatelets (GNP) and amine-functionalized graphene nanocomposites demonstrated enhanced mechanical properties. Amine-functionalized nanocomposites, which were more fragile than raw epoxy, showed improved tensile strength. This results from graphene nanoplatelet aggregation [5].

Accordingly, 2-weight percent GNP nanocomposites demonstrated superior characteristics compared to one-dimensional CNTs in epoxy composites containing functionalized carbon nanotubes [CNTs] and GNPs composite. Due to variations in pull-out processes and the bridging effects of CNTs, the reinforcement effects of two-dimensional GNPs were stronger than those of one-dimensional GNPs. [6] .The fabrication of nanocomposites is greatly influenced by the size, orientation, interaction, and dispersion of nanoparticles. Applications for graphene in mechanical and microelectronic devices are found in its good mechanical and thermal properties [7]. A magnetic composite made of epoxy resin, GO, MWCNTs and Fe3O4 performs better mechanically and thermally thanks to the orientation of the nanoparticles. The results demonstrated an improvement in mechanical properties for 1.5 percent weight of GMF, with bending strength increasing by 136.5 percent and impact strength increasing by 30.9 percent [8]. An epoxy composite loaded with MWCNTs in low concentrations (0.1-0.4 wt. %) exhibited higher strengths under tensile and bending loads. An increase in tensile and flexural by 61 % and 150 % was observed in nanocomposites loaded with 0.3 wt. % MWCNTs, when compared to plain epoxy [9]. Micro (SiC, and Al2O3) and nanofillers (MWCNTs) were used to suppress the initiation of a crack in Glass-Epoxy laminates. Laminates loaded with 0.5 wt. % SiC exhibited higher bending strength and crack suppression was achieved with the filler addition [10].

Many researchers are interested in using natural fibers as fillers in the epoxy matrix because they are less expensive, healthier, and more environmentally friendly than synthetic carbon and glass. Natural fiber composites demonstrated enhanced tensile strength, flexural strength, and toughness [11]. In the literature, it has been discussed the effects of reinforcing natural fibers such as palm, birch, and eucalyptus in an epoxy matrix at a dose of 35 weight percent. The combination of resin transfer molding and molded fiber-based processing approach produced stronger tensile strength with eucalyptus/epoxy among the three kinds when compared to other fibers like palm and birch [12]. Fibers derived from the different parts of the date palm tree were used as reinforcements in epoxy composites. The results showed that the addition of different fibers substantially improved the modulus and strength of the fabricated composite compared to the epoxy composites [13]. Wood flour derived from palm trees was used as reinforcement in Low-density polyethylene composites (LDPE). The fiber-reinforced composites exhibited lower tensile and flexural strength than pristine LDPE, which was attributed to the poor filler–matrix interface [14].

Due to their weather resistance and improved mechanical qualities, epoxy-based matrix composites reinforced with silica nanoparticles (SNs) and epoxidized natural rubber (ENR) as fillers were employed for drone blades [15]. When paired with SN, ENR demonstrated a decrease in glass transition temperature (Tg) and an increase in epoxy resin modulus. The use of silica nanoparticles helped the epoxy matrix become more durable. As the ENR percentage grew, yield strength declined. When epoxy and hybrid SN were combined, yield strength improved as the SN's % grew. Compared to clean epoxy, ENR does not increase the interfacial strength between the matrix and epoxy [16]. In the aerospace industry and as a fire-resistant casing for electrical devices, epoxy-based hybrid composites showed good fire resistance and improved structural qualities [17].

Since synthetic polymer products cannot degrade, their use has dramatically increased, causing environmental pollution and health risks for people. The urgent requirement is for a suitable biodegradable substance that can take the place of polymer composites that are not biodegradable. In this direction, PLA exhibits enormous promise as a substitute for goods derived from petroleum [18]. Environmental pollution can be decreased by a synergistic mixture of biopolymer and synthetic polymer supported by strong interfacial contacts and controlled dispersion. Bio-composite offers a wide range of uses in

the packaging, food processing, medicinal, textile, and electronics industries. PLA, which is created from renewable resources like corn starch and sugarcane, is now a widely utilized biopolymer material.
The current study investigated the effects of graphene and $SiO_2$ addition on the mechanical, and fracture properties of epoxy-PLA nanocomposites. Fractured surfaces from the tensile tests were examined under SEM to understand the effects of filler addition on the tensile strength of epoxy-PLA composites. The influence of filler addition on Tensile strength was also studied using FE simulation.

## EXPERIMENTAL DETAILS

### *Materials*

The matrix used is a blend of epoxy resin and PLA. Epoxy resin (Lapox-L12), which is cured by K6 hardener, was applied in a ratio of 9:1. Polymer products were supplied by Atul India Ltd, Ahmedabad, Gujarat, India, and granules of PLA were procured from Nature Tech India Pvt. Ltd. The filler materials used were Graphene and SiO2, whose particulars are given in Tab. 1 and Tab. 2 respectively.

| Characteristic Property | Value |
|---|---|
| X and Y dimensions | 10-20 microns |
| Length | 3-6nm |
| Purity | 96-99% |
| Number of Layers | The average number of layers 1-3 |
| Tensile strength | >5 GPa |
| Tensile Modulus | >1,000 GPa |

Table 1: Properties of Graphene nanosheets.

| Characteristic Property | Value |
|---|---|
| Particle size | 10-20 nm |
| Density | 2.2-2.6 g/mL at 25 °C |
| Purity | 99.5% |
| Tensile strength | 100 MPa |
| Tensile Modulus | 70 GPa |

Table 2: Properties of $SiO_2$ nanoparticles.

## PREPARATION OF NANOCOMPOSITES

In this work, nanocomposite samples of six different types were prepared. Fig. 1 depicts the procedure followed to prepare nanocomposites. Before ultrasonication, a measured amount of epoxy is heated to reduce its viscosity. After adding the fillers to the ethanol, the solvent was sonicated for 10 minutes. Then, the fillers were added to the epoxy, which was first mixed manually for about 5 minutes before being sonicated for around 50 minutes. A cold-water bath was used to keep the solution temperature under control while sonication was carried at 40 kHz. After sonication, K-6 hardener and PLA solution were added to the filler-epoxy mixture and manually mixed for 5 minutes. PLA solution was prepared by dissolving PLA granules in Tetrahydrofuran (THF) solution [19] with continuous stirring for around 24 hours. Finally, the solution is cast in a mold of 230 mm x 160 mm x 3 mm and cured for 24 hours at room temperature. The cured sample was

taken out of the mold and the samples were cut as per the specifications of ASTM standards. The details of the nanocomposites prepared are given in Tab. 3.

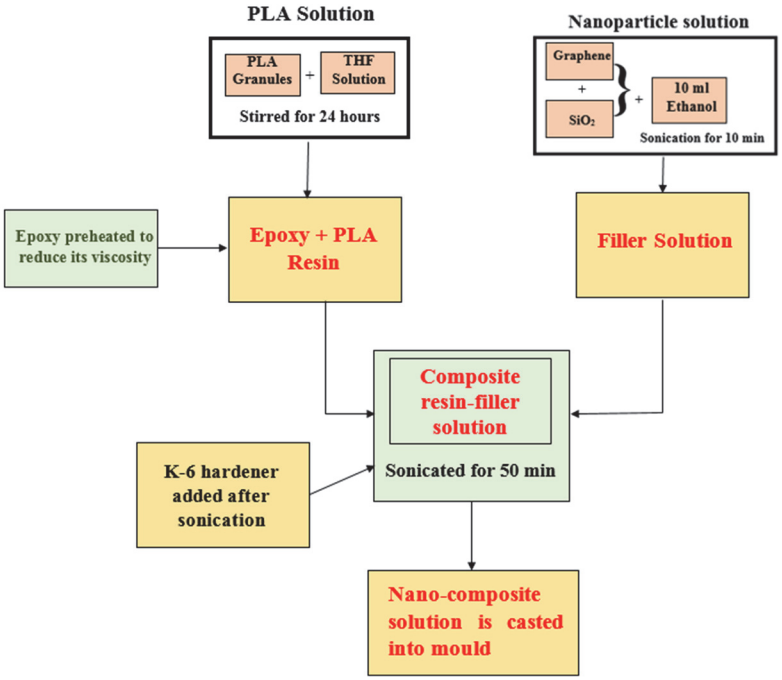



Figure 1: Steps in preparation of nanocomposites.

| Serial No. | Specimen Code | Epoxy, wt.% | PLA, wt.% | Graphene wt.% | $SiO_2$ wt.% |
|---|---|---|---|---|---|
| 1 | S0 | 80 | 20 | 0.00 | 0.00 |
| 2 | S1 | 79.9 | 20 | 0.05 | 0.05 |
| 3 | S2 | 79.8 | 20 | 0.10 | 0.10 |
| 4 | S3 | 79.7 | 20 | 0.15 | 0.15 |
| 5 | S4 | 79.6 | 20 | 0.20 | 0.20 |
| 6 | S5 | 79.5 | 20 | 0.25 | 0.25 |

Table 3: Composition of the pristine epoxy-PLA and nanocomposites

## CHARACTERIZATION TESTS OF COMPOSITES

### *Tensile test*

The tensile strength of all samples was evaluated using a Tinius Olsen universal testing machine with a capacity of 10kN in accordance with ASTM D 638 [20] and a crosshead speed of 3mm/min. Fig. 2 depicts the dimensions of a tensile specimen. Five samples from each category were tested, and the average result was used to calculate the tensile strength of composite samples.

### *Flexural test*

Flexural strength was evaluated using a 3-point bending test according to ASTM D 790 [21] using a Tinius Olsen universal testing machine with a capacity of 10kN and a crosshead speed of 3mm/min. The dimensions of the flexural specimen are

presented in Fig. 3, and an 80 mm span length was chosen. Five samples from each category were examined, and the average result was used to calculate the flexural strength of composite samples.

*Fracture toughness test*

Single-edge notch bend (SENB) specimens were used to determine the fracture toughness ($K_{IC}$) as per ASTM D5045 [22]. The tests were carried out with a crosshead speed of 1 mm/min using Tinius Olsen UTM (10kN Capacity). The specifications of the specimen are shown in Fig. 4 with the support length being 48 mm. $K_{IC}$ was obtained by using Eqn. (1):

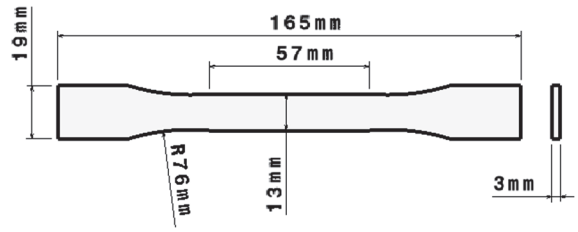



Figure 2: Tensile test specimen.

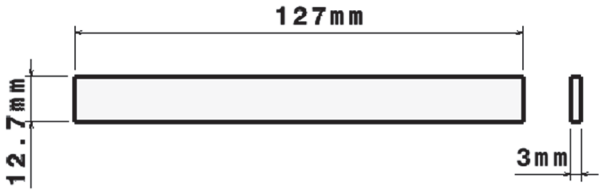



Figure 3: Flexural test specimen.

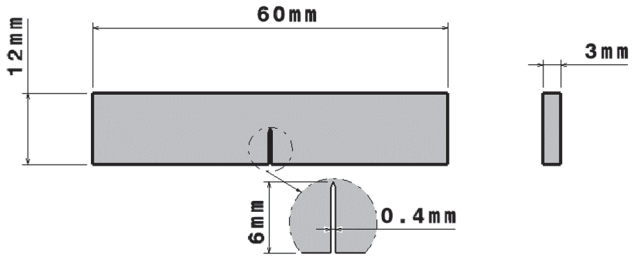



Figure 4: SENB specimen.

$$K_{IC} = \left( \frac{P_Q}{BW^{1/2}} \right) f(x)$$

where $0 < x < 1$ and

$$f(x) = 6x^{1/2} \frac{\left[ 1.99 - x(1-x)(2.15 - 3.93x + 2.7x^2 \right]}{(1+2x)(1-x)^{3/2}}$$

where $P_Q$– Load, kN; $B$- Thickness of specimen, cm; $W$- Width of specimen, cm; $a$- Length of crack, cm; $x = a / w$

## RESULTS AND DISCUSSIONS

*Tensile test results*

The stress-strain curves of the pristine PLA/Epoxy resin and resin loaded with $SiO_2$-Graphene fillers are shown in Fig. 5. Tensile strength of all composite specimens determined from the stress-strain curves is summarized and presented in Fig. 5. The tensile strength of two-phase composites was substantially affected by the filler concentration. The filler concentration significantly affected the tensile strength of nanocomposites. The tensile strength of S0 is 23.07 MPa; it can be observed that as the proportion of the hybrid fillers $SiO_2$ and graphene increased in the holding matrix from 0.1 to 0.3 wt. %, the tensile strength also increased from 29.61 MPa to 36.86 MPa. When the filler concentration is increased above 0.3 wt. %, the tensile strength decreased significantly in the range of 13.60 MPa to 17.83 MPa. This reduction might be due to nanoparticle aggregation in the matrix. This decrease can be linked to nanoparticle clustering in the matrix.

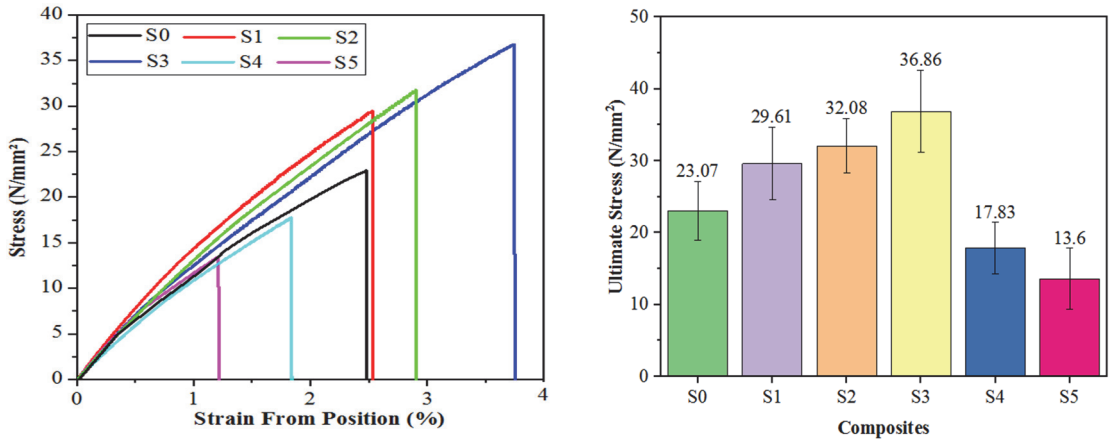



Figure 5: Stress-strain curves (left) and Tensile strength (Right) of all composites

*Flexural test results*

The flexural strength of all laminates obtained from the bending strength is summarized in Fig. 6. It was found that the fillers used had a significant impact on the flexural strength of composites. The bending strength of the plain Epoxy-PLA composite was 51.7 MPa, as the concentration of fillers increased from 0.1 to 0.3 wt. %, flexural strength improved from 67.46 MPa to 81.03 MPa. As the filler concentration increased beyond 0.3 wt. %, a drastic drop in flexural strength was observed, composites loaded with 0.4 and 0.5 wt. % fillers exhibiting flexural strength of 54.36 MPa and 46.15 MPa respectively. Graphene may tangle and agglomerate at higher concentrations due to its high aspect ratio and van der Waals attractive contacts, reducing the bending strength.

*Fracture toughness test results*

The fracture toughness of the pristine Epoxy-PLA and filler-loaded samples are shown in Fig.7. Neat composite exhibited a fracture toughness of 1.8 MPa.m1/2. As the filler concentration increased from 0.1 to 0.3 wt. %, an increase in toughness in the range of 22.83 % - 97.26 % was observed. As the concentration of fillers is further increased, a reduction in fracture toughness was observed, with S4 and S5 exhibiting a 30.18 % and 37.05 % reduction in toughness respectively compared to S3. This phenomenon can be attributed to the agglomeration of nanoparticles with higher concentrations of filler content.

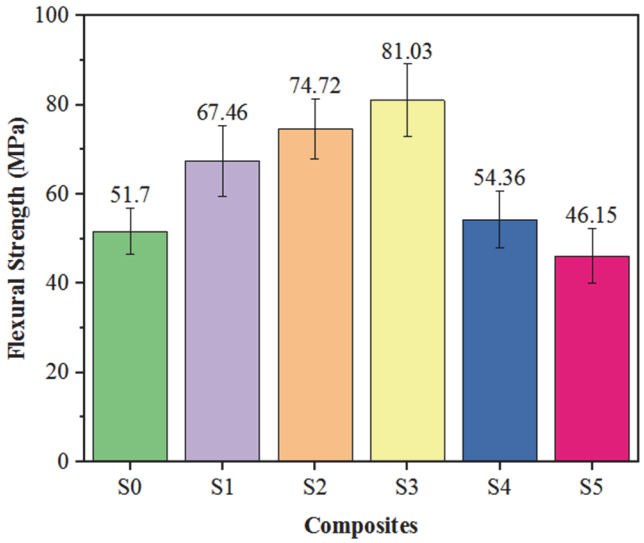



Figure 6: Flexural strength of all composites

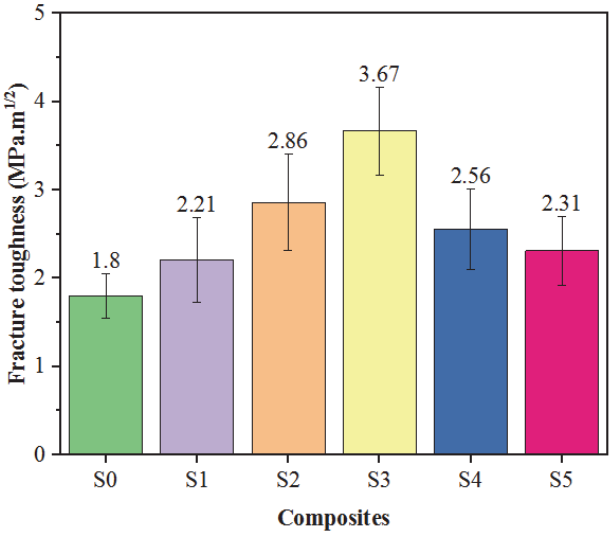



Figure 7: Fracture toughness of all composites

*Analysis of SEM images*

The fractured surfaces from the tensile tests were examined using the SEM, to understand the effects of filler addition on the interfacial interactions of the filler matrix and the degree of dispersion [23-25]. For the pure epoxy-PLA sample, Fig. 8(a) depicts an extremely smooth surface, which implies the brittle mode of fracture. In Graphene-$SiO_2$ loaded epoxy-PLA

samples, from Fig. 8(b)-(d), cleavage planes and surface roughness can be seen. In the case of filler-loaded samples, the nano-sheet structures can be seen along with an increase in the roughness of the surface. As the filler concentration increased to 0.4 wt. %, an agglomeration can be observed in Fig. 8 (e), which resulted in the decline in tensile strength.

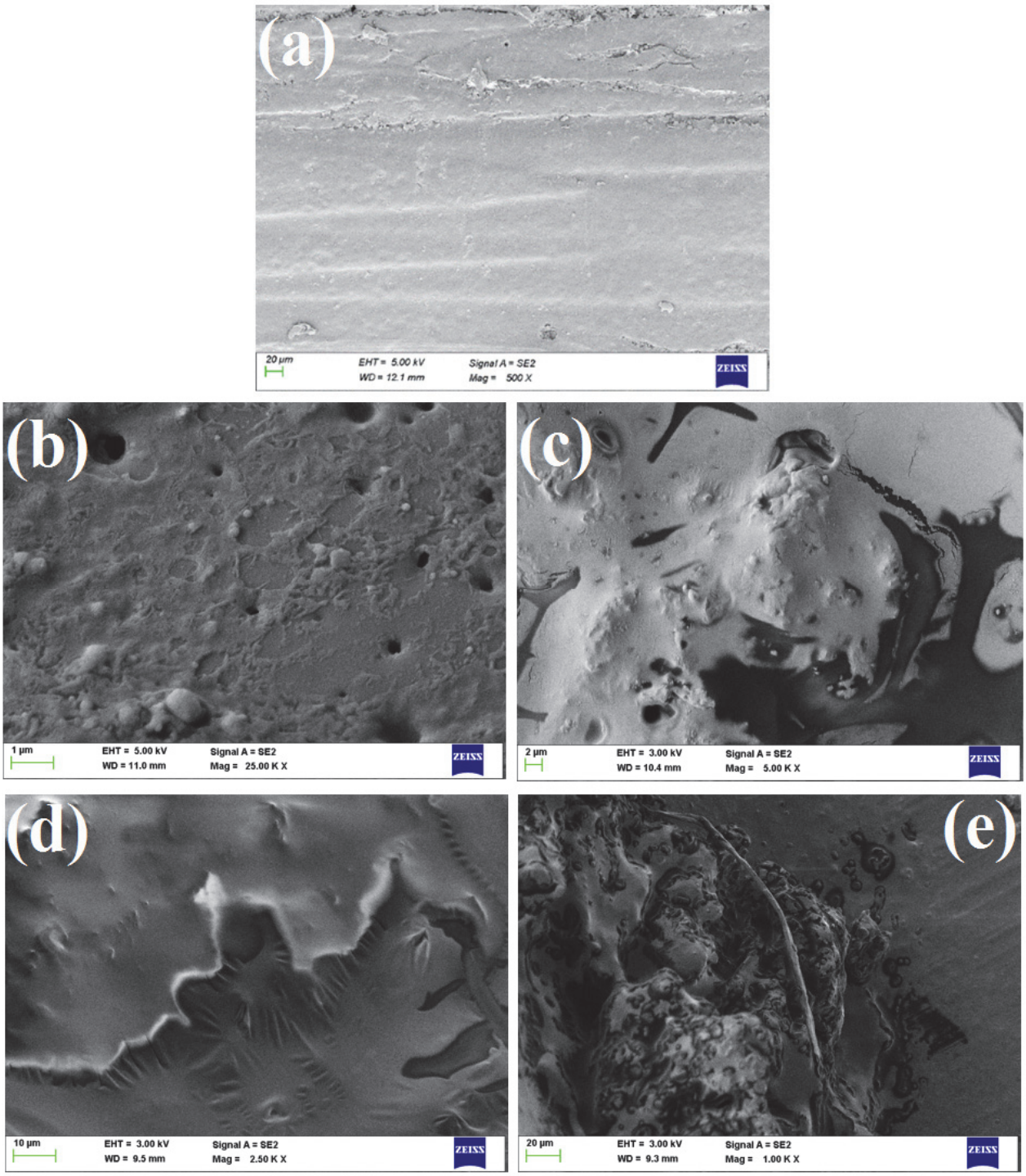



Figure 8: SEM micrographs of fracture surfaces from tensile tests of (a) S0 (b) S1 (c) S2 (d) S3 (e) S4.

### *Simulation studies*

Simulation studies involve Finite element analysis to predict the mechanical properties obtained and the same is validated using the experimental results.

*Creation of material and model*

In the Material Designer module of ANSYS Workbench, new material was created under the Engineering Data section, which is shown in Fig. 9(a). The basic properties like Young's modulus, Poisson's ratio, and ultimate strength obtained from the tensile test were used to create matrix material (20% PLA + 80% epoxy resin). Other elasticity properties such as bulk modulus and shear modulus were derived from the available inbuilt method in the material designer. RVE [26-27] was created for each nanocomposite with Epoxy-PLA as a matrix and with varying proportions of Graphene and $SiO_2$ as reinforcements. The mechanical properties of the Graphene (Tab. 1) and $SiO_2$ (Tab. 2) were used to create the RVE. Fig. 13 shows meshed RVE. This material developed is taken as input for structural analysis in ANSYS Workbench. The 3D model for tensile test analysis was modeled in Solid works using the component model, the model was created as per ASTM D638. These models were precise replicas of the proportions of the specimens used for experimentation.

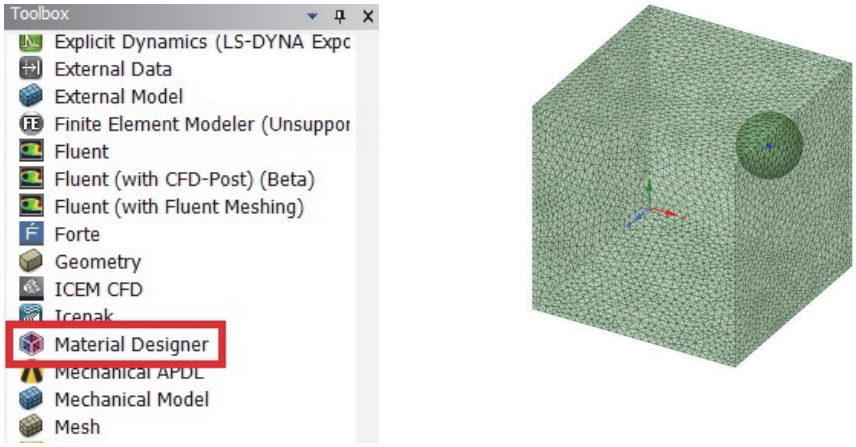
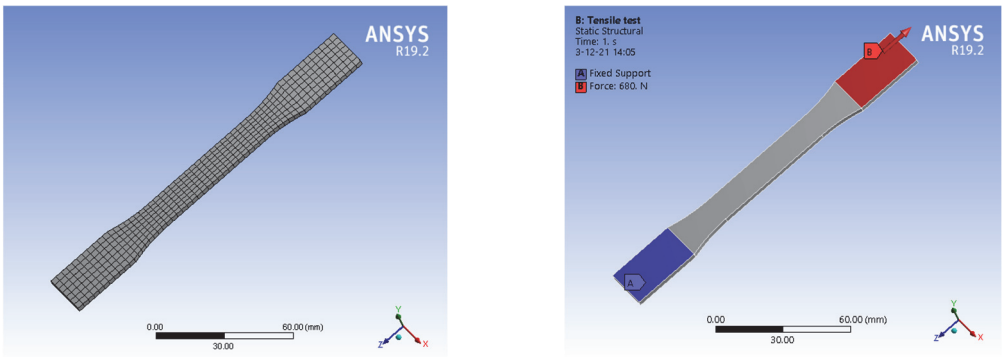



Figure 9: (a) Material Designer module (b) RVE created in Material Designer.

## Mesh Generation and Boundary Conditions

The tensile model was meshed with Solid 92 elements as shown in Fig. 10(a). To simulate tensile tests, one end of the specimen is clamped and axial force was applied on the other end as shown in Fig. 10(b). Ansys Explicit Dynamics was used to solve the meshed model and generate the results. Lastly, the properties of the solver; initial conditions, system statics, dynamic properties, and desired output were defined.




(a) (b)

Figure 10: (a) Meshed tensile specimen (b) Tensile specimen with applied boundary conditions.

## Simulation Results

The tensile strength obtained from the simulation of tensile samples using ANSYS Explicit Dynamics and the experimental data is enlisted in Tab. 4. Fig. 11 shows the variation of stress in the S3 specimen. The S3 specimen exhibited higher tensile strength as presented in Tab. 4. The tensile strength for the S3 specimen obtained from the

simulation and experimentation were 40.63 MPa and 36.86 MPa respectively. The variations in the tensile strength obtained from the experimentation and simulation for all nanocomposites were within 20 %.

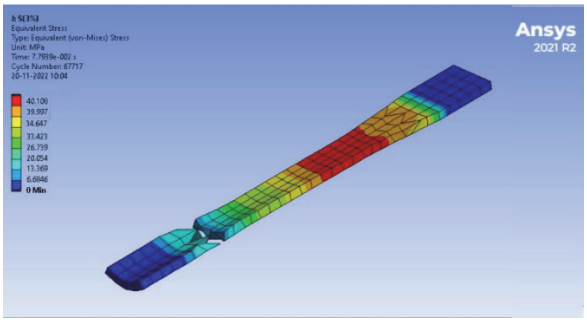



Figure 11: Maximum equivalent stress in the S3 tensile specimen.

| Specimen | Tensile strength from simulation in MPa | Tensile strength from experimental results in MPa | Variation in results (in %) |
|---|---|---|---|
| S0 | 26.54 | 23.07 | 15.04 |
| S1 | 32.46 | 29.61 | 9.63 |
| S2 | 33.36 | 32.08 | 3.99 |
| S3 | 40.63 | 36.86 | 10.23 |
| S4 | 20.65 | 17.83 | 15.82 |
| S5 | 16.1 | 13.6 | 18.38 |

Table 4: Simulation results for the samples developed.

## Conclusions

This work investigated the effects of Graphene and $SiO_2$ on the mechanical and fracture properties of Epoxy-PLA nanocomposites. The composites were manufactured with varying contents of Graphene and $SiO_2$ (0.1-0.5 wt. %) by solution casting. The composites loaded with 0.3 wt. % fillers exhibited higher tensile, flexural, and fracture toughness compared to other nanocomposites. An increase in tensile strength in the range of 29 % to 60 % was observed with the addition of fillers from 0.1 to 0.3 wt. %, further addition of fillers resulted in a drastic decrease in tensile strength. The flexural strength of composites improved by 5 – 57 % with the addition of nanofillers. An improvement of fracture toughness in the range of 23-104 % was observed in hybrid nanocomposites in-comparison to pristine epoxy-PLA composite. It can be concluded that epoxy-PLA nanocomposites loaded with both Graphene and $SiO_2$ exhibited better mechanical and fracture properties compared to pristine epoxy-PLA composites. The tensile strength of nanocomposites determined through experimentation and numerical analysis were in close agreement.

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