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Flexural Properties of Graphene Reinforced Polymer Nanocomposite Synthesized by Stereolithography

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Abstract

A new era of production has been ushered in by the recent advancement of additive manufacturing (AM) techniques in a number of industrial areas, including biomedicine, building, electronics, telecommunication, mechanical, and defense. The design and manufacture of customized parts can be done with more freedom thanks to additive manufacturing (AM) technologies, which also promote quick production, waste reduction (and, in some circumstances, elimination), a decreased risk of human mistake, high precision, and accuracy at reasonable prices. The mechanical strength, thermal conductivity, and electrical conductivity of polymers are inferior than those of metals and alloys, which are the three main criteria for choosing materials in a variety of applications. In order to create polymer composites with an infinite variety of types and properties, these properties can be enhanced by adding the appropriate additives to polymers. Combining the polymer composites' limitless flexibility with additive manufacturing (AM) fabrication enables the production of goods that are quick, affordable, effective, and multifunctional. The current work focuses on the creation of graphene reinforced polymer nanocomposites and the mechanical characterization of those materials. The findings of the experiment show a considerable improvement in flexural strength upto 38.1% from 13.12MPa (G-0wt %) to 18.12MPa (G-0.5wt %), highest flexural strength reaches 26.74MPa (G-1.5wt %).

Keywords: Additive manufacturing; Mechanical properties; Thermal conductivity; Graphene; polymers

1. Introduction

An extensive amount of research has gone into creating polymer nanocomposites, which employ hard, nanofiller with a high aspect ratio, Carbon nanotubes (CNT) or clay are two examples, to enhance the mechanical, thermal, or other properties of the polymer. Comparing polymers to metals and alloys reveals that they have less desirable mechanical, thermal, and electrical characteristics, Electrical conductivity, mechanical toughness, and thermal which are important factors when choosing materials for different purposes. These qualities may be enhanced by using the right additives, and polymer composites are available in an infinite variety of forms and with a wide range of characteristics. We can enhance the mechanical, thermal, and electrical qualities of components by combining the nearly limitless flexibility of polymer composites with additive manufacturing [1-4].

The idea of employing graphene, a new addition to family of carbon molecules, as reinforcement for several polymer matrices has been suggested. Graphene has a sheet-like structure and is composed of a single layer of one's orbital and two p orbital's carbon atoms. It also possesses outstanding physical qualities and a low density. It has been demonstrated via a number of theoretical and experimental research on graphene that it has a large specific surface area as well as superior mechanical, thermal, and electrical characteristics. Because of its excellent electrical and thermal conductivity, graphene has potential use in heat sinks, housing and electronic circuit boards, battery cells, and motherboard fins [5].

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Among various technologies, the 3D printing has made significant advancements over the past ten years among the many industrial processing methods. The key advantages of this technology over standard prototype techniques are increased precision, faster speed, and reduced material waste. Additionally, a 3D printer doesn't impose any limitations on the product's form and doesn't call for any extra equipment. There are numerous varieties of 3D printers on the market right now, and they make use of a number of different technologies, such as Laminate Object Manufacturing (LOM), Stereo lithography (SLA), Digital Light Processing (DLP), Fused Deposition Modeling (FDM) and Selective Laser Sintering (SLS) [6-9].

Stereolithography apparatus (SLA), a 3D printing technology, provides the highest printing resolution capabilities when compared to other 3D printing technologies [10]. SLA is a technique that can be applied to medical equipment such as patient-specific scaffolds and dental implants because photo-polymerization is used to aggregate the layers in SLA. In order to produce SLA products with high resolution and structure precision, precise space and time control of the applied photons is necessary [11]. For Stereolithography, which frequently uses epoxy- or acrylate-based materials, the original resin is constrained by photo polymerization, which to turn limits the process. The process of making photosensitive resin is still time-consuming and costly, but recent investigations have shown that some synthetic materials may also be used in SLA. Blending with nanocomposites is a good choice to strengthen and functionalize the SLA-printed items [12]. For example wood flour was demonstrated by Shuyang Zhang et al. to increase the tensile strength of composites that are SLA produced [13]. Although Chi Him Alpha Tsang et al [14] used graphene oxide nanocomposites. Oriented nanoclays were reinforced by H Eng et al. to improve mechanical properties [15]. Methacrylate/chitin nanowhiskers composites were reinforced by Reymarj D et al for mechanical and thermal properties [16].Sandeep Kumar et al. reinforced cellulose nanocrystals to boost their mechanical qualities; the mechanical properties were improved by the addition of minute amounts of CNCs [17].Adnene Sakly et al. developed a unique quasicrystal reinforced to enhance mechanical qualities for various industrial applications [18].Using stereolithography, Yukako Sano et al. demonstrated how to create materials with short fibers or continuous fiber-composites to increase mechanical characteristics [19].On the basis of these facts, SLA may be a viable technique for producing complex shapes, and graphene may be contribute for improving the mechanical performance.

In the light of the above, current investigation focuses on synthesis of graphene reinforced polymer based nanocomposites conducted by Stereolithography, evaluation of physical and mechanical properties of SLA based polymer nanocomposites and to compare with unreinforced SLA parts and to characterize its flexural strengthwith varied percentage of Graphene.

2. Materials and methods

2.1 Materials

Black Liquid Anycubic UV Resin 405nm is made up of 600g with epoxy acrylate, 200g with Hexamethylene diacrylate bifunctional monomer (HDDA), and 200g of trifunctional monomer trimethylolpropane triacrylate as its primary constituents (TMPTA). HDDA has a viscosity between 5 and 10 cps, as per commercial recommendations, which might thin out the entire system and lessen the creation of bubbles. Figure 1 depicts the SEM of the graphene used to create the composite. The results demonstrated that the graphene utilised was multilayered and in sheet form. Energy Dispersive Spectroscopy was also utilised to examine graphene. The EDS result for graphene powder is depicted in Figure 2.

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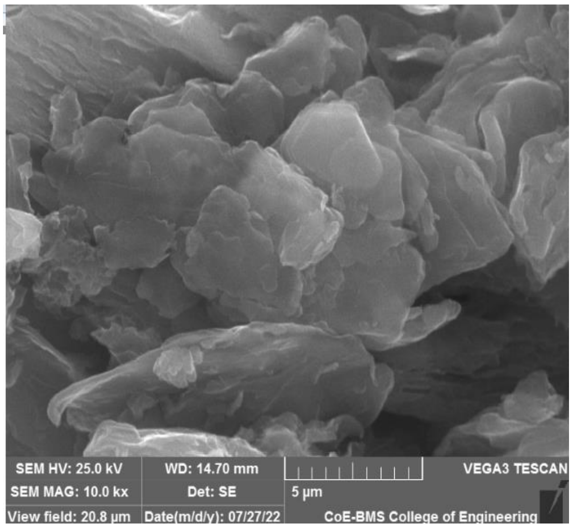

Fig. 1: SEM of Graphene

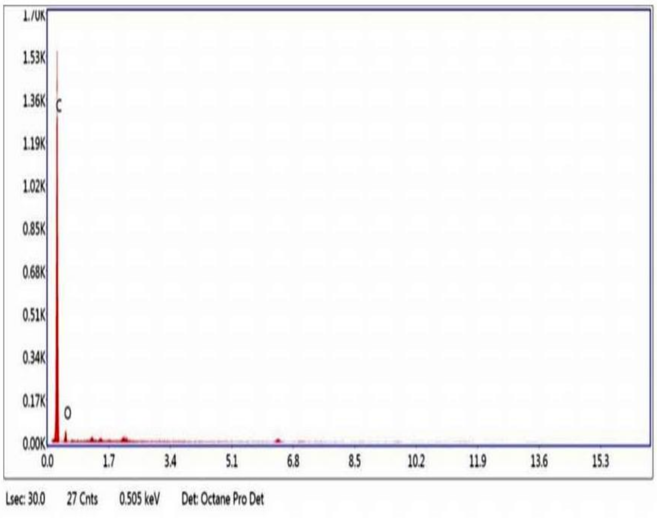

Fig. 2: EDS of Graphene

2.2 Preparation of Graphene-Polymer resin

The standard black Anycubic resin (99.5g) is taken in a round glass beaker, and then the Graphene powder (0.5g) was added to the resin to form 0.5wt% graphene/resin mixture solution and magnetically stirred for 2 hours at a speed of 1300Rpm for uniform distribution of graphene powder in resin, after the stirring process the mixture is taken immediately for printing. Similarly for 1wt %, 99g of resin, 1g of graphene is taken and for 1.5wt%, 89.5g of resin, 1.5g of graphene is taken.

2.3. 3-Dimensional printing of the composites

The Solidworks software is used to build the stereolithography files (.stl) for the necessary specimen. After that, photon slicing software was used to slice the imported stl files. The objects were manufactured using a stereolithography printer at a temperature of 25°C with 0.1mm-thick layers. You can find further printing specifications in the accompanying data, such as the layout and support structures. Using a manual process, the samples were removed from the printing plate after printing. The samples were progressively submerged in solutions of isopropyl alcohol for five min each to eliminate any remaining unprocessed resin from the surface. The specimens were then allowed to air dry for a one day before further use. The samples were given the identifier G-x, where x stands for the percentage of graphene added. For example, G-1 denotes a composite containing 1 percent of graphene. Furthermore, the printed samples without graphene were labeled as G-0.

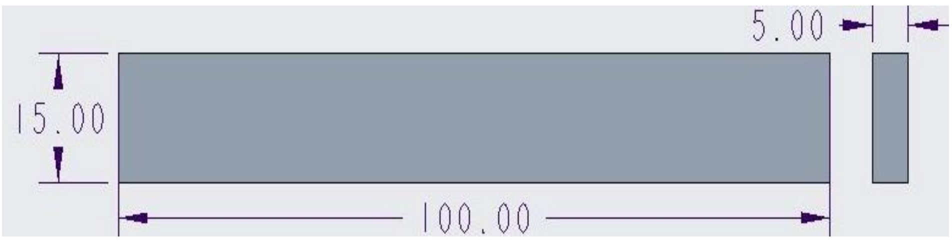

Fig. 3: CAD model image (all dimensions are in mm)

2.4. Characterization of printed samples

2.4.1 Dimensional accuracy

Dimensional accuracy is one statistic that may be used to assess the quality of final products produced by additive manufacturing. Accuracy is a measurement's resemblance to its actual value. In 3D printing actual value is equivalent to the CAD-designed dimensions.The measurements of the part's features were measured using a Electronic Vernier caliper (Mitutoyo) once printing was finished. The linear dimensions were measured three times, and the average measurements are computed. To determine the dimensional correctness, the dimensions were compared with the actual dimension.

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2.4.2 Surface roughness

In this research, the roughness of the specimens' surfaces was assessed using the contact technique. For this purpose, the Mitutoyo Surface Roughness Tester is used. The specimen length (Lr) was chosen to be 2.5mm in order to increase measurement accuracy because the roughness of surface of Stereolithography specimens was between 2<Ra<10. On each surface, the roughness measurements were carried out five times, the average result was given as the surface roughness of the intended specimen.

2.4.3 Flexural strength

Flexural strength and modulus are most typically measured using flexural testing. The maximum stress at the outermost fibre on either the compression or tension side of the specimen is termed as flexural strength. Rectangular cross-section specimens were printed according to ASTM D7264 with dimensions of 100 15 5 mm were created for the three-point bending flexural test. The total number of work piece for flexural test is 4 including 0wt% materials as shown in figure 4.

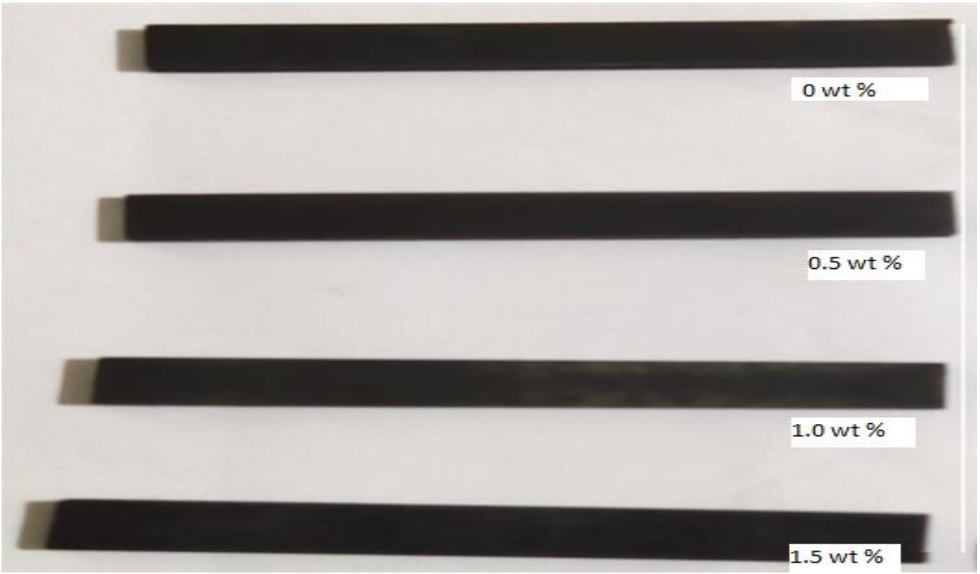

Fig. 4: Flexural test samples

3. Result and discussion

3.1 Surface roughness

The specimen length (Lr) was chosen to be 2.5mm in order to increase measurement accuracy because the roughness of surface of SLA specimens was in the range of 2<Ra<10. On each surface, the roughness measurements were executed five times, and the average outcome was reported in Table 1.

Table 1: Average Surface roughness of printed samples

Samples	G-0wt%	G-0.5wt%	G-1wt%	G-1.5wt%
Average values	2.786μm	1.286μm	1.27μm	1.266μm

When comparing the roughness of different specimens, the roughness (Ra) average values are shown in the graph in Figure 5. The average values for roughness of surface are shown in Table 1. The figure 5 demonstrates how decreasing the amount of graphene leads to a smoother surface. Because of the reinforcement's dimensional stability and homogeneous distribution, the surface polish value may have improved. Homogeneous distribution makes it feasible to enhance both heat conductivity and dimensional stability.

Surface roughness has decreased by 53.8%% when 0.5 wt% of graphene is added to a pure resin sample. Similar to how the increase trend is followed, so is the decrease trend. In compared to the addition of 0.5 wt% Graphene, in addition to 1.0 wt% graphene sample resulted in a reduction of 1.024%. Similar to this, 1.5 weight percent of

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graphene added resulted in a decrease of 0.78% as opposed to 1.0 weight percent. The addition of 1.5 weight percent of graphene reinforcement results in the largest reduction, which is 54.55% when compared to the pure resin sample (G-0 wt %).

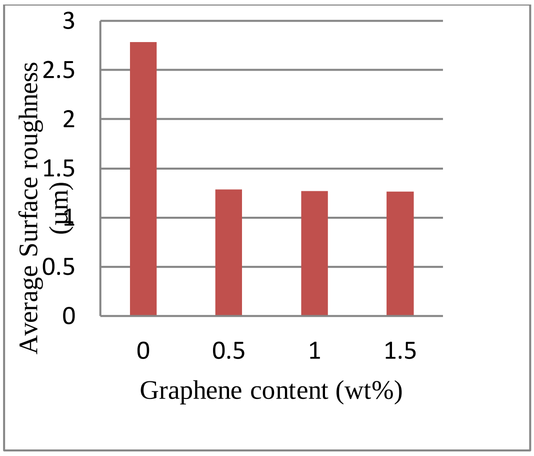


Fig.5: Surface roughness of printed samples

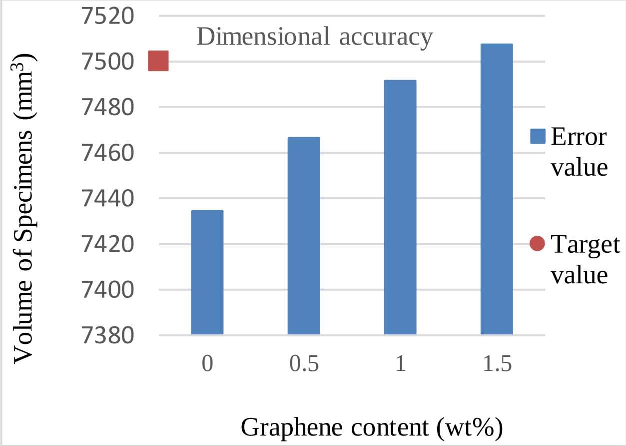


Fig. 6: Dimensional accuracy of printed samples

3.2 Dimensional accuracy

The measurements of the part's features were measured using an Electronic Vernier caliper (Mitutoyo) once printing was finished. The linear dimensions were measured three times, and the average measurements are computed in Table 2. To determine the dimensional correctness, the dimensions were compared with the actual dimension. The actual dimension is a designed dimension. The dimensions of designed sample are 100mm length, 15mm width and 5mm thickness

Table 2: Dimensions of printed samples

Material	**Dimensions**		
	Length, mm	**Width, mm**	**Thickness, mm**
G-0wt%	100.06	15.01	4.95
G-0.5wt%	100.09	15.04	4.96
G-1wt%	100.09	15.06	4.97
G-1.5wt%	100.10	15.06	4.98

Figure 6 Shows the comperssion of dimensions of printed samples with designed sample. The obtained results clearly tells that, the all printed samples have dimensional error. But the reinforced samples (G-0.5 wt% G-1 wt% and G-1.5 wt%) are more accurate than the pure sample (G-0 wt%). Graphene with a 1.5 weight percent content is more accurate than other samples.

3.3 Flexural strength

Figure 7 displays the bending strength of printed samples with various percentage of Graphene (G-0wt%, G-0.5wt%, G-1 wt%, and G-1.5wt %). It is observed that graphene reinforced (G-0.5wt%, G-1wt%, and G-1.5wt %) nanocomposite exhibit superior Flexural strength in comparison to unreinforced (G-0wt %). The flexural strength for pure resin (G-0 wt %) sample is 13.12Mpa. With the increase in the concentration of just 0.5 wt% of Graphene, there is substantial improvement in the flexural strength of the nanocomposite material, as shown in Figure 7. By adding 0.5 wt% of Graphene, the improvement in flexural strength of the material is from 13.12MPa to 18.12MPa. At 1 wt% Graphene content, the flexural strength of the composite material increases up to 24.82MPa, At 1.5 wt% Graphene content, the flexural strength of the composite material increases up to 26.74MPa. There is a 103.80% increase than the pure sample. According to Figure 7, there is a gradual increase in the flexural strength of the nanocomposite material with an increasing concentration of Graphene. This confirms the observations reported in the work that the addition of graphene facilitates the enhancement of the flexural strength.

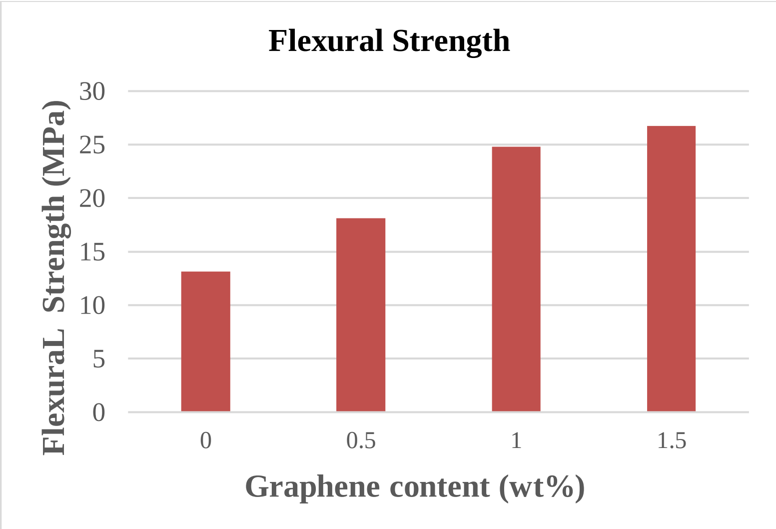
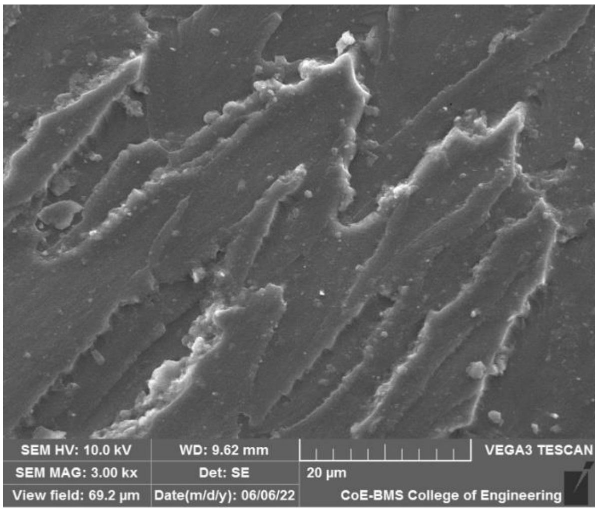


Fig. 7: Flexural strength of printed samples

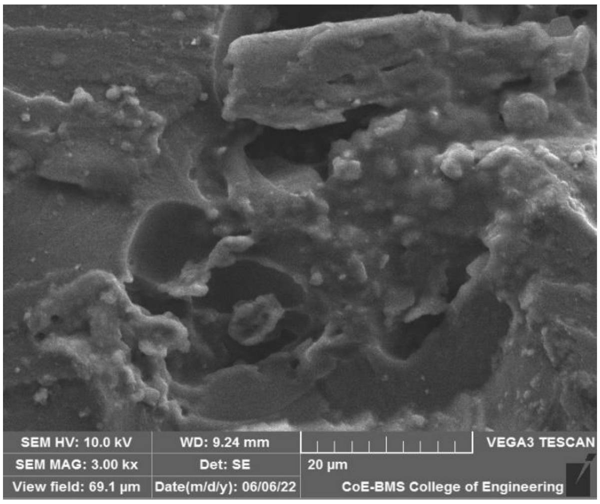
3.4 SEM analysis of fractured surface

Figure 8 displays the SEM of the broken specimens obtained from flexural testing. To demonstrate how the use of graphene lowers the amount of voids, the samples were printed using the SLA technique. The physical properties of the blended nanocomposite are improved by appropriate mixing and dispersion.

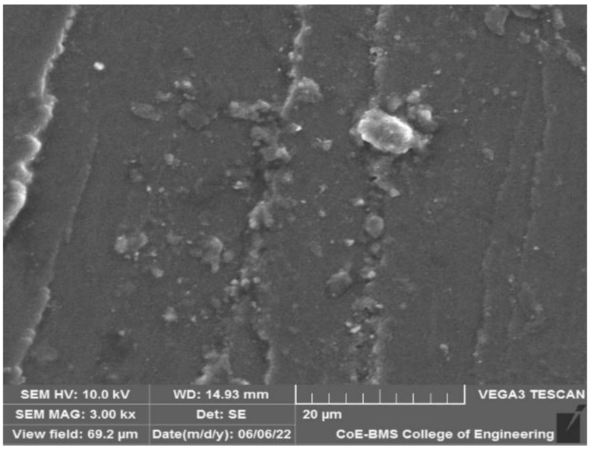
When comparing nanocomposite samples to pure samples, we can see that the ductile dimples are less common in the nanocomposite samples. This suggests that the amount of graphene increases the brittleness of the nanocomposite by decreasing its ductility.



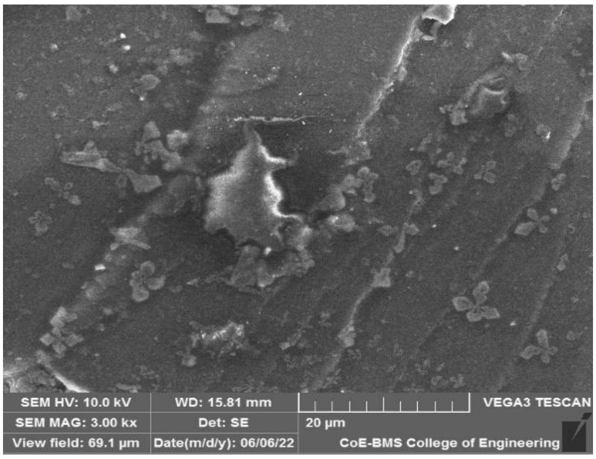
A) G-0wt%



B) G-0.5wt%



C) G-1wt%



D) G-1.5wt%

Fig. 8: SEM imageries of flexural fracture samples A) G-0wt% B) G-0.5wt% C) G-1wt% D) G-1.5wt%

4. Conclusions

In this current study, carbon based polymer nanocomposite were fabricated using stereolithography based additive manufacturing. Main conclusions are listed as follows,

- The resin/ graphene mixtures are readily mixed by magnetic stirring process.
- Composites specimens with 0.5, 1 and 1.5 wt % are successfully printed by Stereo lithography technique.
- Surface roughness of graphene content samples were decreased compared to samples without graphene.
- The dimensional accuracy showed that, all printed samples have dimensional error. But the reinforced samples are more accurate than the pure sample. Graphene containing 1.5 wt% is more accurate than other samples.
- Compared to the sample without graphene, the printed graphene-incorporated composites had increased flexural strength. After addition of graphene, the flexural strength was improved up to 38% from 13.12 MPa to 18.12MPa (0.5 wt% graphene) while the highest flexural strength was obtained as 26.74MPa (1.5 wt% graphene).

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