



Nanohybridization Effects on Impact Behavior of Basalt/Glass Fiber Reinforced Epoxy Hybrid Nanocomposites

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ABSTRACT

Basalt fiber-based composites have received greater attention from manufacturers and researchers recently for use in a variety of structural applications, owing to their high strength-to-weight ratio, high stiffness, and excellent mechanical properties. The main objective of the current research is to examine the impact behavior of hybrid basalt/glass fiber reinforced composites that have been altered by the addition of MWCNTs and SiO₂ (Nano silica) to the epoxy matrix as per the ASTM standards. All of the composites were created using Manual lay-up method followed by Compression molding using novel symmetric stacking sequence configurations of B/GG/BB/GG/BB/GG/B fibers with nanoparticles at weight percentages of 0%, 1% (0.5% MWCNTs + 0.5% SiO₂) 2% (1% MWCNTs + 1% SiO₂) and 3% (1.5% MWCNTs + 1.5% SiO₂). The homogeneous dispersion of nanoparticles in the epoxy matrix was achieved by ultrasonicator and magnetic stirrers. The maximum value of Izod impact strength was recorded as 203 kJ/m² for the composite filled with 2 wt. % of nano-fillers. In comparison to the composite containing no fillers, the composite adding 2 wt. % showed a 24% improvement from 167 to 203 kJ/m² in Izod impact resistance. It was observed that increasing the weight percentage of fillers in composites causes insufficient interfacial interaction between the matrix and fibers, thereby decreasing their impact resistance. The load transmission between the matrix and fibers is increased by the presence of MWCNTs/SiO₂ at the fiber/matrix interface, which also improves the fracture toughness. According to images obtained by Scanning Electron Microscopy of the fracture surfaces of impact-tested specimens, the major reasons for failure include fiber/matrix debonding, fiber breaking, fiber fracture and fiber pull-out.

Keywords: Hybrid composite; Basalt fiber; Glass fiber; Nano fillers; Impact strength.

1. INTRODUCTION

In recent years numerous researchers are working in the area of hybrid nanocomposites where two or more reinforcements are used to reinforce a base material. The main objective of hybrid composite is to overcome the drawbacks of single reinforcement composite. The fibers are considered as primary reinforcement such as natural fibers (jute, kenaf, bamboo, bagasse, coir, banana, etc.) and synthetic fibers (glass, carbon, aramid fiber (Kevlar), etc.); whereas, particles are considered as secondary reinforcement used in the form of micro and nano sizes. Besides, Kevlar and carbon fibers are more expensive than basalt fiber which is obtained from volcanic rocks originating from frozen lava; basalt fiber is the best replacement for carbon fiber because of its strength, stiffness and low cost (Lebedev *et al.* 2020). Furthermore, basalt fiber is a natural mineral fiber possessing excellent mechanical strength, superior temperature resistance, chemical resistance and corrosion resistance properties; it can be used as an alternative to carbon fiber (Abd El-baky *et al.* 2020). Despite this, glass fiber has been the most commonly

used reinforcement in composites for the past five decades because of its low cost; it is a sustainable material with good mechanical, corrosion, moisture and chemical resistance properties. Moreover, hybrid composites find many applications which include aircraft, automotive, marine and construction industries (Raajeshkrishna *et al.* 2020). In addition to fibers, various fillers such as TiO₂, Al₂O₃, Nano clay, Nano silica, and CNTs were added to the polymer matrix to further enhance properties. The fillers between the matrix and fibers improve the interfacial bonding and act as barriers to crack propagation (Li *et al.* 2014). Furthermore, Al₂O₃, SiO₂, and TiO₂ microparticles were added to the epoxy matrix to improve the mechanical properties of the glass fiber/epoxy composite. In comparison to other micro-modifiers, it has been found that SiO₂-treated epoxy composites have higher flexural strength, flexural modulus, and interlaminar shear strength (ILSS). This might be a result of silica's smaller particle size compared to other materials (Nayak *et al.* 2014).

Nagaraja *et al.* (2020) reported the influence of stacking arrangement on the tensile and flexural

characteristics of laminated carbon-glass/epoxy hybrid composites made using the resin infusion technique. This composite laminate with outer glass fabric revealed interface decohesion as a significant failure mechanism and demonstrated the significance of stacking carbon fabric in the correct location to get improved tensile and flexural performances.

Abd El-Baky *et al.* (2022) developed hybrid composites comprised of flax, basalt and E-glass fibers by using vacuum bagging technology, and their mechanical characteristics were examined. Compared to flax fiber/epoxy composite, these hybrid composites had better tensile, flexural and impact properties. The relative amounts of the fibers and the order of stacking have noticeable effects on the mechanical performance.

Dhand *et al.* (2015) reviewed basalt fibers as a substitute for glass fibers when used as a reinforcement material for composites. Globally, there has been a recent growth in the use of natural, eco-friendly fibers as reinforcement in the production of inexpensive, lightweight polymer composites. Basalt fiber is one such material of interest that is being utilized widely; it is affordable and provides outstanding characteristics more than glass fibers.

Arshad *et al.* (2021) examined how basalt fiber-reinforced bio/synthetic epoxy hybrid composites' physico-mechanical and thermal properties were affected by coir fiber and titanium carbide (TiC) nanoparticles. A tensile fracture state analysis and the flexural, tensile, impact, porosity, and water absorption tests were performed. With the effect of the highest load transmission between the fillers and matrix materials, the inclusion of coir fiber and TiC nanoparticles demonstrated a considerable improvement in mechanical and thermal characteristics.

Mishra *et al.* (2020) evaluated the influence of adding graphene to glass fiber epoxy composites at various weight percentages, including 0% (S1), 1% (S2), 2% (S3), and 3% (S4). Due to its novelty, affordability, and compatibility with other polymers, graphene offers remarkable qualities including high absolute strength and aspect ratio; it has outstanding potential as a nano filler. On glass/epoxy Nano composite laminates with two different lay-ups [(0/90)12s and (0/90/45)6s], the flexural, tensile, and impact tests were conducted. In view of these findings, graphene has the potential to be a useful reinforcement in polymer composites due to its ability to significantly enhance a variety of mechanical properties of glass fibre epoxy composite when combined with the parent material.

Mittal and Rhee (2018) analysed, on basalt fabric, CNTs were grafted using chemical vapour deposition (CVD). Additionally, basalt fabric that had been grafted with CNTs (BF-CNT) was sandwiched

between epoxy utilizing the hand lay-up technique. By using XRD, HR-RAMAN, FE-SEM, and thermo gravimetric analysis (TGA), the BF-CNT was characterised. The characteristics of the created BF-CNT/epoxy composites were also studied, and they were compared to those of BF/epoxy composites reinforced with CNTs.

Petrucci *et al.* (2013) utilising glass, flax, and hemp fibre laminates to create symmetrical configurations with all of them having a 21-23% fibre volume in an epoxy resin, the various hybrid composite laminates were created by vacuum infusion, and the properties of each were compared. The mechanical performance of each hybrid laminate appears to be better than basalt fibre laminates and inferior to pure hemp and flax fiber-reinforced laminates. The hybrids with the highest properties are those made from basalt fiber-reinforced laminates with glass and flax added.

Raajeshkrishna *et al.* (2019) prepared Sandwich hybrid composites by using the hand layup technique and then compressed them. The sandwich composites were made using glass and basalt textiles and epoxy resin. The fabrics were utilized both naturally and after being treated with sodium hydroxide and hydrochloric acid. According to the findings, surface treatment and hybridization increase the composites' tensile strength and hardness. The sandwich composites with a glass fabric skin and a basalt fabric core that were subjected to hydrochloric acid treatment had the highest tensile strength.

Ricciardi *et al.* (2019) experimentally investigated the impact of stacking order on the impact damage mechanisms and matrix-fiber dependent parameters such as flexural and interlaminar strength, of epoxy hybrid basalt/flax composites. While the flexural modulus showed no influence from the fiber order, the flexural and interlaminar shear strengths varied depending on the resin concentration. The impact tests were conducted at various energy and penetration levels. A confocal microscope was used to quantify the indentation caused by a hit to learn more about the features and damage propagation that were influenced by the type of hybridization and the impact energy.

Sapuan *et al.* (2020) examined the hybrid composites made of longitudinal basalt and woven glass fibers reinforced with unsaturated polyester resin. The mechanical properties of hybrid composites were improved by the combination of glass fiber with basalt. By hand laying up the unsaturated polyester resin (UP), basalt (B), and glass fibers (GF) in six different formulas (UP, GF, B7.5/G22.5, B15/G15, B22.5/G7.5 and B), the composites were made. This study demonstrated that basalt boosted the density, tensile and flexural properties of glass-fiber-reinforced unsaturated polyester resin.

Erkliğ *et al.* (2020) manufactured the hybrid glass/basalt fiber composites containing nanographene (GnP) to study the properties of low velocity impacts at 30 J of impact energy. Additionally, their tensile and flexural characteristics were experimentally characterized. Both laminate configurations, including basalt and glass at the outer and inner skins, were subjected to impact and mechanical tests during the studies. The study's findings showed that the impact and mechanical properties significantly improved with the addition of GnP at 0.1 weight percent, demonstrating improved load transfer between GnP-matrix resin-fiber interactions.

Megahed *et al.* (2019) reported the effect of silica and carbon nanofillers added to epoxy reinforced with woven and nonwoven tissue glass fiber. It examined how varied weight fractions of carbon black and silica nanoparticles affected the tensile characteristics, impact strength and fatigue performance of epoxy matrix reinforced with two distinct types of E-glass fiber. In comparison to pure glass fiber reinforced epoxy composites (NGFRE), the results revealed a significant improvement in tensile characteristics, impact strength, and fatigue life with the inclusion of practically all nanoparticle concentrations.

Sanjay *et al.* (2018) studied the impact and inter-laminar strength of E-glass with jute/kenaf woven fabric epoxy composites to assess the impacts of hybridization on various laminate stacking sequences constructed with jute, kenaf, and E-glass textiles using the vacuum bagging method. The findings demonstrate how glass fabrics can be added during hybridization to improve the characteristics of jute/kenaf textiles-reinforced epoxy composites. Jute/kenaf fabric hybridization with E-glass fabric hybridization offers a way to outperform pure natural fiber-reinforced composites in terms of mechanical impact and inter-laminar strength. When compared to conventional laminates, the hybrid laminate with jute fiber plies as the core layer and skin layers made of E-glass and kenaf fibre plies as the skin layers performed better.

Among these, the contribution of Megahed *et al.* (2019) is closely related to this research work.

From the literature survey, it was evident that very limited research work had been done on particulate-reinforced hybrid polymer composites. It is planned to produce hybrid composites reinforced with hybrid nano fillers to cover this research gap and better understand the mechanical behavior of the newly created hybrid material. The manufacturing and characterization of hybrid glass and basalt fiber reinforced epoxy composites filled with MWCNTs and SiO₂ (Nano silica) were thoroughly investigated, and the findings were presented in this paper. This study's novelty originates from the combined effect of basalt/glass fiber hybridization with a

novel stacking sequence (B/GG/BB/GG/BB/GG/B) and MWCNTs/SiO₂ inclusions on impact qualities such as absorbed energy and maximum load-bearing. Combined MWCNTs/SiO₂ fillers of weight contents (0%, 1%, 2% and 3%) were used for this purpose, along with basalt/glass hybrid configurations. A thorough investigation has been done on the benefits of using MWCNTs/SiO₂ simultaneously added in basalt/glass fiber-reinforced composites.

2. MATERIALS AND METHODS

2.1 Materials Used

Table 1 presents the specifications of E-Glass and Basalt fabrics. Figures 1 (a) and 1 (b) displayed the Plain weave E-glass fiber (bi-directional) with a specific surface area weight - 200 g/m², 300 x 300 tex (warp and weft yarn specification), and width 100 cm, and plain weave basalt fiber (bi-directional) with a specific surface area weight - 200 g/m², 132 tex (warp and weft) and width 100 cm and also epoxy resin (Araldite LY556) with a density of 1.20 gm/cc at 25 °C and hardener (Aradur HY951) with a density of 0.98 gm/cc at 25 °C as shown in Fig. 1 (e) were supplied by Hayael Aero Space India Pvt. Ltd., India. The epoxy and hardener were combined in a 10:1 ratio to make the matrix system. The chemical compositions of basalt and glass fiber are presented in Table 2.

Secondary reinforcements for hybrid composites included nano silica (SiO₂) particles with an average particle size of 20-50 nm and multi-walled carbon nanotubes (MWCNTs) with outer and inner diameters were 10-30 nm and 5-10 nm respectively, as shown in Figures 1 (c) and 1 (d). These nanoparticles were supplied by AD Nano Technologies Pvt. Ltd., Bangalore, India. The image of the E-Glass/Basalt fabrics and nanoparticles is shown in Fig. 1. The fiber weight percentage was 60%, as recommended by Sanjay *et al.* (2018).

Table 1. Specifications of used E-Glass and Basalt fabrics

Parameters	E-Glass fabric	Basalt fabric
Woven style	Plain	Plain
Density (gm/cc)	2.70	2.55
Surface area weight (GSM)	200	200
Moisture content (%)	0.06	0.10
Thickness (mm)	0.48	0.20
Width (mm)	1000	1000
Warp yarns (yarns/m)	300	132
Weft yarns (yarns/m)	300	132

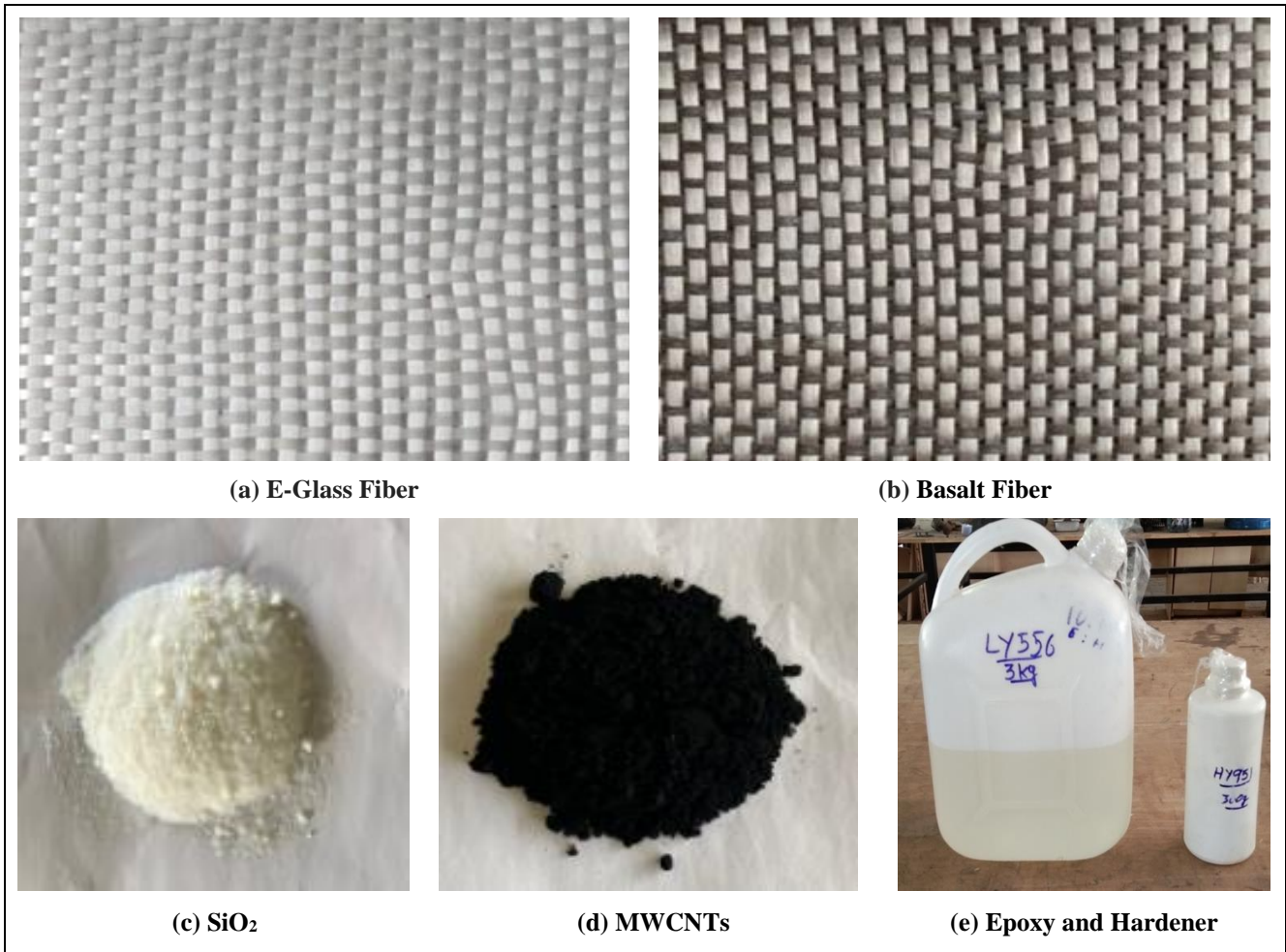


Fig. 1: Materials used for fabrication (a) E-Glass fiber (b) Basalt fiber (c) SiO₂ (d) MWCNTs and (e) Epoxy and Hardener

Table 2. Chemical Composition of E-glass fiber and Basalt fiber

Fibers	Major components (wt. %)						
	SiO ₂	Al ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	Fe ₂ O ₃ + FeO
Basalt fiber	48.8 - 57.5	14 - 18.2	5.2 - 10	1.3 - 16	1.9 - 6.4	0.8 - 4.5	4.0 - 13.3
E-Glass fiber	52 - 56	12 - 16	16 - 25	0 - 5	0 - 2	0.2 - 0.8	<0.3

Table 3. Composition of Basalt/Glass fibers and fillers used in hybrid composite fabrication

Laminate code	Novel stacking sequence	Fiber (wt. %)	Fillers (wt. %)	Matrix (wt. %)	Thickness (mm)
BG1 (0%)	B/GG/BB/GG/BB/GG/B	60 %	0%	40%	3.0
BG2 (1%)	B/GG/BB/GG/BB/GG/B	60 %	1% (0.5% MWCNTs + 0.5% SiO ₂)	39%	3.2
BG3 (2%)	B/GG/BB/GG/BB/GG/B	60 %	2% (1% MWCNTs + 1% SiO ₂)	38%	3.1
BG4 (3%)	B/GG/BB/GG/BB/GG/B	60 %	3% (1.5% MWCNTs + 1.5% SiO ₂)	37%	3.3

2.2 Fabrication of Nanocomposite Laminates

Several steps were used to prepare the composite samples. First, the mold surface is coated with a release and adhesive agent (wax) to prevent epoxy from adhering to the surface. A thin plastic sheet is then put to the top and bottom of the mold plate to create a flat surface for the composite laminates. 6 layers of E-glass woven and 6 layers of basalt woven for a total of 12 layers were used as reinforcement in each composite and were cut to the required size of 300 x 300 mm and placed on the surface of the mold plate. The used basalt/glass fibers and their filler composition in the fabricated hybrid nanocomposites are presented in Table 3. The ultrasonicator was then utilized to homogeneously mix MWCNTs and SiO₂ nanofillers with predetermined weight percentages in epoxy resin for 20 minutes. After the ultra-sonication process, the magnetic stirrer was used to mix the epoxy system with the hardener in a 10:1 ratio for 10 minutes.



Fig. 2: Impact testing machine setup (Izod Test)

The modified resin was now poured onto the surface of the fiber that was already in the mold and applied evenly with a brush. The other mats are subsequently placed on top of the prior epoxy layer and pressed with a roller to eliminate any trapped air bubbles and excess resin. This process was continued until all of the 12 layers were placed one over the other. The mold plate was then placed on the top surface of the plastic sheet and uniform pressure was applied with the help of concentrated weights over the mold plate which is kept at the same pressure for 24 hours at atmospheric temperature to cure the composites according to the materials supplier's guidelines. After 24 hours of curing at room temperature, the mold is opened, and the woven

composite is removed from the mold surface. According to ASTM standards, all test composite samples were cut using a computerized, numerically controlled abrasive water jet machine. The composite laminate manufacturing process was carried out by hand lay-up method followed by compression molding.

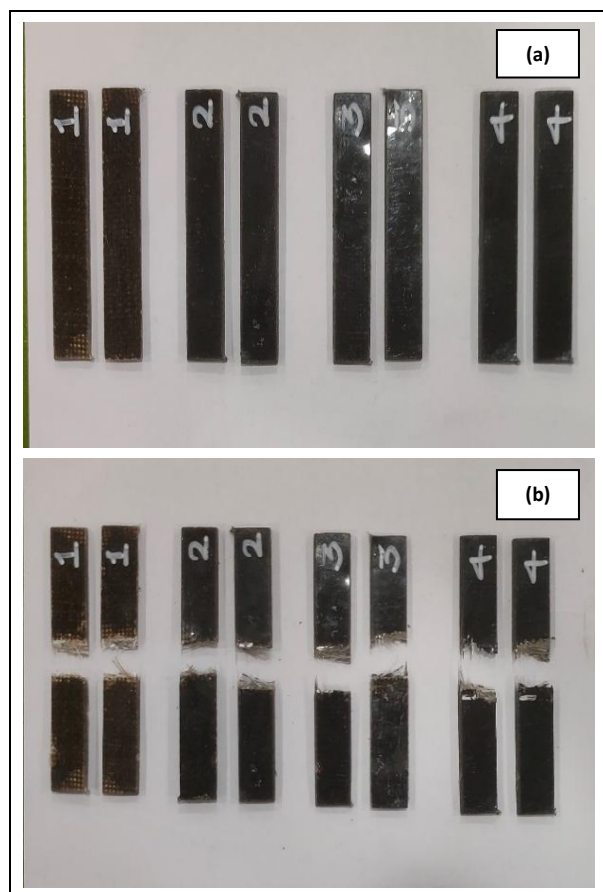


Fig. 3: Impact specimen images (a) before Impact test and (b) after Impact test

2.3 Mechanical Characterization

2.3.1 Impact test

Using the Izod test, as per ASTM 256, the impact strength and absorbed energy were assessed (Santhosh *et al.* 2018). Impact tests were performed using a 25.4 J impact testing machine (Brand: International Equipment) shown in Fig. 2. The impact strength of the specimen is calculated as the energy absorbed per unit area of the fractured cross-section. The toughness and ductility of the specimen were measured by recording the amount of energy absorbed before it cracked, in terms of J/m. It shows that brittle materials only absorb a small amount of energy whereas tough materials absorb a lot. At room temperature of 25 °C, the test was conducted on all samples. Figures 3 (a) and 3 (b) show specimens of impact test before and after testing.

3. RESULTS AND DISCUSSIONS

3.1 Impact Properties

A material's capacity to absorb energy at fracture when subjected to a sudden impact is demonstrated by an impact test. Fig. 4 depicts the impact strength after the nanoparticles are added to the epoxy matrix. The impact characteristics of manufactured composite samples are presented in Table 4. It can be seen from these data that the average impact strength was measured at 167, 203 and 163 kJ/m², respectively, which is higher than the impact strength of a composite having no fillers, which was recorded at 157 kJ/m². This is because the epoxy matrix contains MWCNTs + SiO₂ which increases fracture toughness. Because particles are filled in various geometrical shapes, the interfacial bonding between the matrix, particles and fibers significantly increases. The composite having 2 wt. % of fillers displays the maximum value of impact strength as displayed in Fig. 4. Thus, it has been found that increasing the nanoparticles results in a drop in the impact strength value.

Table 4. Impact properties of fabricated composite samples

Laminate code	Impact Strength (KJ/m ²)	Impact Energy (J)	Thickness (mm)
BG1 (0%)	157	5.2	2.60
BG2 (1%)	167	5.4	2.70
BG3 (2%)	203	7.9	2.65
BG4 (3%)	163	5.5	2.75

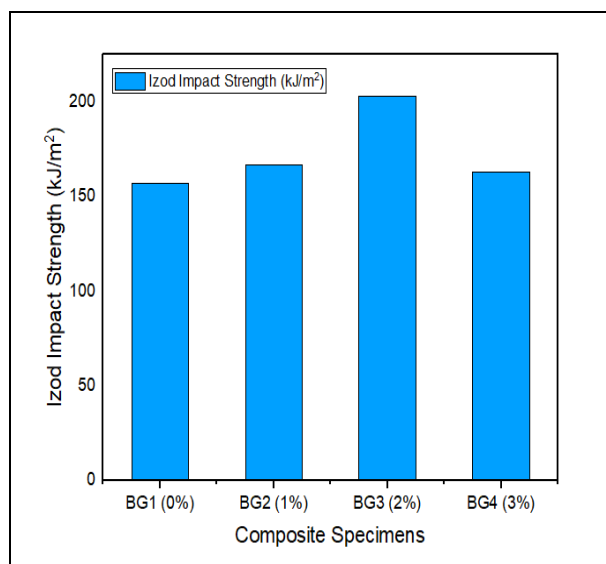


Fig. 4: Variation of Impact strength (kJ/m²) for different wt. % composite samples

Nanoparticle-filled matrix provides more effective stress transfer, which in turn lowers the local

stress concentration near the interlayer of fiber and the matrix. This enhances the mechanical properties and interfacial adhesion of Nanocomposite laminates Tian *et al.* (2017). Nanoparticles can function as interlocking pins in the interphase and create more friction between the matrix and fiber, strengthening the interfacial adhesion. When bonded to a polymer matrix, nanoparticles' great strength and stiffness make them useful for carrying heavy loads (Hosur *et al.* 2017). The impact energies were recorded as 5.2, 5.4, 7.9 and 5.5 J for unfilled and filled composites of BG1 (0%), BG2 (1%), BG3 (2%) and BG4 (3%), respectively and presented in Table 4. From this, it was observed that an impact energy of 2 wt. % of filled composite has maximum energy than the others shown in Fig. 5. This is due to better interfacial bonding between matrix and fibers in the composite.

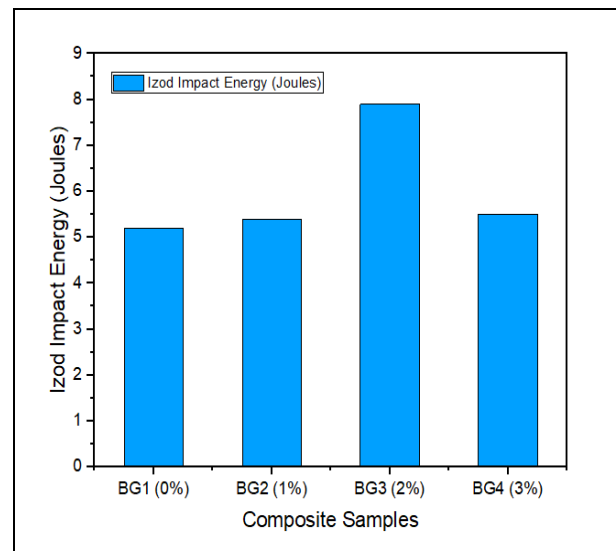


Fig. 5: Variation of Impact strength (kJ/m²) for different wt. % composite samples

3.2 SEM Analysis

Fig. 6 shows the SEM micrographs of specimens at the fractured surfaces of hybrid composites made of basalt/glass fiber-reinforced and filled with MWCNTs + SiO₂, which were taken after the impact test. The specimen without MWCNTs/SiO₂, as shown in Fig. 6 (a), has smooth-surfaced individual fibers, indicating weak adhesion between the fiber/matrix interface regions., Figures 6 (b) and 6 (c) show rough surfaces on the fibers, when MWCNTs/SiO₂ are added to the composites.

This may be characterized by the enhanced adhesion between the fiber/matrix interactions at the epoxy matrix, which results in enhanced fracture toughness. The fiber/matrix interfacial interactions are relatively weak, as seen by Fig. 6 (c), where some of the fiber surfaces have little matrix sticking to them.

Furthermore, agglomeration of particles in the matrix has been found, thereby reducing bonding between the BG4 (3%) specimen's fibers and matrix (Megahed *et al.* 2019).

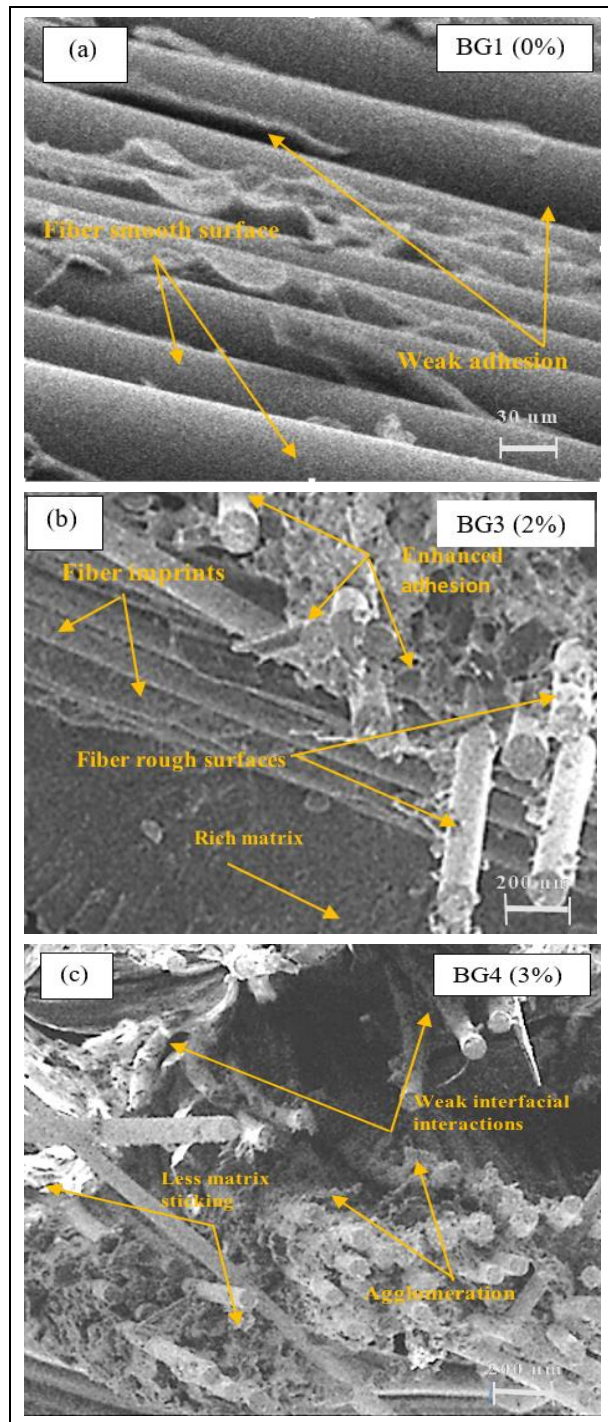


Fig. 6: SEM micrographs of Impact test specimens at fracture surfaces for (a) BG1 (0%), (b) BG3 (2%) and (c) BG4 (3%) composite laminates

4. CONCLUSIONS

The mechanical characteristics of the epoxy matrix, reinforced with basalt/E-glass fibers, multiwalled

carbon nanotubes and nano-silica were studied. Epoxy resin was simultaneously infused with nano-silica and multi-walled carbon nanotubes. To achieve proper dispersion of these reinforced nanoparticles in epoxy resin, an ultrasonicator and magnetic stirrers were used to embed them into the epoxy. The synthesized hybrid nanocomposites might be appropriate for automobiles, aircraft, and aerospace bodies. Under impact loadings, these hybrid nanocomposites were tested according to ASTM standards. Based on the results obtained, the following conclusions were drawn:

- When compared to unfilled composites, hybrid nanocomposite filled with 2 wt. % (1.0% MWCNTs + 1.0% SiO₂) produced the highest improvement of 24% impact strength.
- For a hybrid composite specimen filled with 2 wt. % produced the maximum impact energy of 7.9 J than other filled and unfilled specimens.
- The addition of a small quantity of nanoparticles enhances more impact characteristics of filled composites when compared to unfilled basalt/glass epoxy composites.
- It is very clear that increasing fillers in the epoxy matrix will decrease the mechanical properties of hybrid nanocomposites.
- It has been observed that the fillers' geometrical shape, size and quantity increased the interfacial adhesion between the matrix/particles/fibers in the hybrid composite.
- SEM images revealed evidence of various modes of failure of hybrid nanocomposites.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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