

High Velocity Impact Response of Basalt Fibers/Epoxy Composites Containing Graphene Nanoplatelets

E. Kazemi-Khasragh*, F. Bahari-Sambran, M. Hossein Siadati, R. Eslami-Farsani

Faculty of Materials Science and Engineering, K. N. Toosi University of Technology, Tehran, Iran

*Corresponding author: ela.kazemi.714@gmail.com

Abstract

The effects of adding surface modified graphene nanoplatelets (GNPs) in various weight percentages (0, 0.1, 0.2, 0.3, 0.4, 0.5 with respect to matrix) on the high velocity impact response of basalt fibers/epoxy composites were evaluated. High speed mechanical stirrer and ultrasonic waves were used for the dispersion of GNPs in the epoxy matrix, and hand layup method was utilized for the fabrication of the composite samples. High velocity impact testing was performed using a conical projectile. The results demonstrated that the maximum improvement in the impact limit velocity and energy absorption occurred in the 0.3 wt.% GNPs nanocomposite, i.e., 11 and 23 %, respectively. Also, the electron microscopy studies revealed that the addition of GNPs contributed in improving the impact properties by influencing the matrix and thus enhancing the interfacial characteristics between the basalt fibers and the matrix.

Keywords: Polymer nanocomposites, Basalt fibers, Graphene nanoplatelets, Surface modification, High velocity impact

1. Introduction

Polymer matrix composites (PMCs) are the right choice for use in automotive, aerospace, transportation and other industries due to their exclusive properties such as high strength and stiffness to weight ratio, fatigue resistance, corrosion resistance and low production cost [1-3].

Basalt fiber is a natural (mineral) fiber and due to its superb properties, it has found a special place alongside the glass and carbon fibers [4]. Basalt is an igneous rock which forms as a result of freezing of alkali magma near the earth's surface [5,6]. Basalt fibers production is similar to that of the glass fibers except that glass fibers are made from molten glass, but basalt fibers are produced directly from the basalt ores. Silica and alumina are the major components of the basalt fibers. Crystallization growth during the basalt fibers production depends on the solidification rate, and changes in the composition of the components lead to variation in their mechanical and physical properties [7-10].

With the advent of nanotechnology, considerable progress has emerged in the manufacturing of nanoparticles to generate new nanocomposites with extraordinary properties [11-13]. One of the most important reinforcements used in nanocomposites is graphene nanoplatelets (GNPs). Graphene is a two-dimensional nanomaterial consisting of pure carbon atoms in the family of carbon allotropes. Owing to its exclusive properties such as thermal, electrical and mechanical, graphene has become an ideal reinforcement candidate for making nanocomposites [14-17].

Shokrieh et al. [18] examined the effect of GNPs on the flexural strength of epoxy/GNP composite. A maximum improvement of 12 % was achieved at 0.25 wt.% GNPs and the addition of 0.5 wt.% GNPs lowered the flexural strength. A study by Nurddin et al. [19] revealed that dispersion of 0.2 wt.% GNPs caused 22.77 % improvement in the flexural

strength. The behavior of glass fibers/epoxy in three-point bend test showed maximum improvement at 0.25 wt.% GNPs [20]. The influence of 0.3 wt.% GNPs increased the tensile modulus of chitosan-epoxy composites by 200 %, but inadequate distribution at higher GNPs decrease it [21]. Chen et al. [22] expressed that 0.1 wt.% GNPs improved the tensile strength of composites up to 3.5 %.

Most studies on fibrous composites reinforced with GNPs have focused on the static properties such as tensile and bending, however, little attention has been paid to their behavior in dynamic loading. The aim of this investigation is to first synthesize nanocomposites by dispersion of various amounts of surface modified GNPs in basalt fibers/epoxy composites, and then evaluate their mechanical properties using high velocity impact testing.

2. Experimental

2.1. Materials

Epoxy resin (KER 828) with polyamine hardener (HA11), from the KUMHO P-B Chemicals Co., Korea, were employed for the matrix material. Atlas weave basalt fabrics with an areal density of 300 g/m², from Basaltex Co., Russia, were used as reinforcement. Graphene nanoplatelets, from US Nano Co., USA, were used as nanometer reinforcement. For surface modification of GNPs, silane coupling agent (4-aminopropyltrimethoxysilane), from Merck Chemical Co., Germany, was exploited. Some properties of the materials used in this study are exhibited in Table 1.

Table 1. Some properties of materials used in this study

Material	Property (unit)	Magnitude
KER 828 epoxy resin	Density (g/cm ³)	1.16
	Dynamic viscosity, ambient temperature (Pa·s)	12-14
	Boiling point (°C)	200
	Flash point (°C)	200
Basalt fabrics	Wave type	Atlas
	Thickness (mm)	0.19
	Areal density (g/m ²)	350
	Yarn density (ends/cm)	
	Warp	15.0
	Weft	7.5
	Breaking load (N/25 mm)	
	Warp	3800
	Weft	1900
	Yarn denier (tex)	68-600
GNPs	Thickness (nm)	2-18
	Length (μm)	4-12
	Purity (%)	95

2.2. Surface Modification of GNPs

2.5 g of GNPs were dispersed in a 100 ml solution consisting of 95 ml ethanol and 5 ml distilled water, using a probe sonicator for 10 min. Then, 2.5 ml of silane agent was added to the dispersion. Surface modification of GNPs in a heated reflux system was performed at a

temperature of 80 °C for 8 hours. It is worth noting that the pH level of the solution was regulated in the range of 4-5 by hydrochloric acid. For separation of GNPs from the silane agent, centrifugal treatment was performed at a speed of 4000 rpm for 30 min and the GNPs were washed with ethanol three times and were held in oven at 80 °C for 24 h.

2.3. Fabrication of Nanocomposites

Silanized GNPs with different weight percentages (0, 0.1, 0.2, 0.3, 0.4, 0.5 with respect to matrix) were added to the epoxy resin. After the initial mechanical stirring (20 min at 2000 rpm) of the GNPs/epoxy resin mixture with a mechanical stirrer, ultrasonic waves were applied to the mixture for 90 min using a probe of 14 mm tip diameter, at 120 W (SDS-11D, Fintek Co., Korea). For elimination of air bubbles, degasification of the mixture was done in vacuum. Finally, the hardener was added to the GNPs/epoxy mixture for curing. The weight ratio of hardener to mixture was 10:100. This mixture was used in fabricating nanocomposite samples containing 6 layers of basalt fabrics (40 % volume fraction) by the hand lay-up method [23]. After 7 days of complete curing at room temperature, mechanical tests were performed on the samples.

2.4. High Velocity Impact Test

The high-velocity impact test was carried out on the composite samples by means of a gas gun (model M211) which could measure the residual velocity. The schematic of gas gun is shown in Figure 1. For high velocity impact test, a cylindrical projectile made out of commercial aluminum with conical nose was used. The mass of the projectile was 27 g. Composite samples had dimensions of 13×13 cm².

The position of composite samples when exposed to the impact load is shown in Figure 2. Moreover, the high velocity impact test was performed with at least five samples and the

average of results was reported. The velocity at which the probability of penetration and stoppage of projectile is equal, V_{50} , and the amount of dissipated projectile kinetic energy, E_{abs} (energy absorption by the composite samples) were calculated using equation (1) and (2) [25]:

where m_p , V_i and V_r are referred to as projectile mass, initial velocity and residual velocity, respectively. It is assumed that the projectile experiences no plastic deformation during the impact.

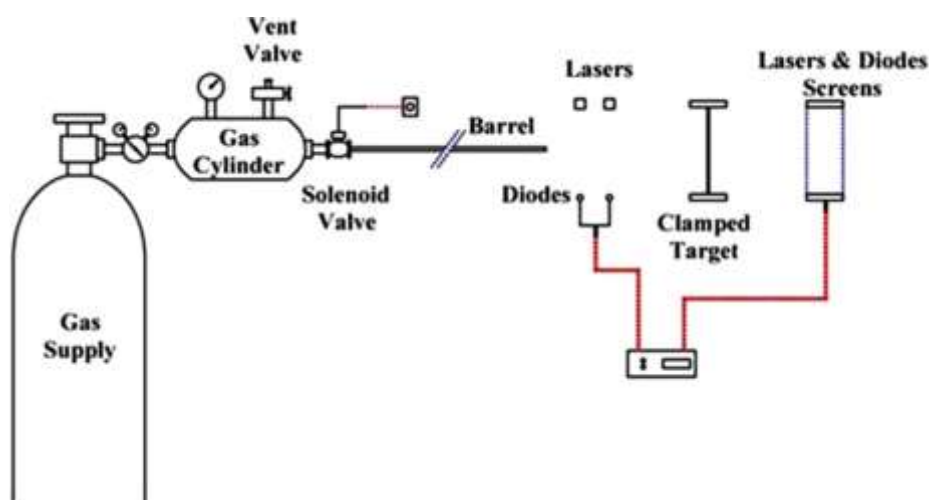


Figure 1. Schematic of gas gun [24].

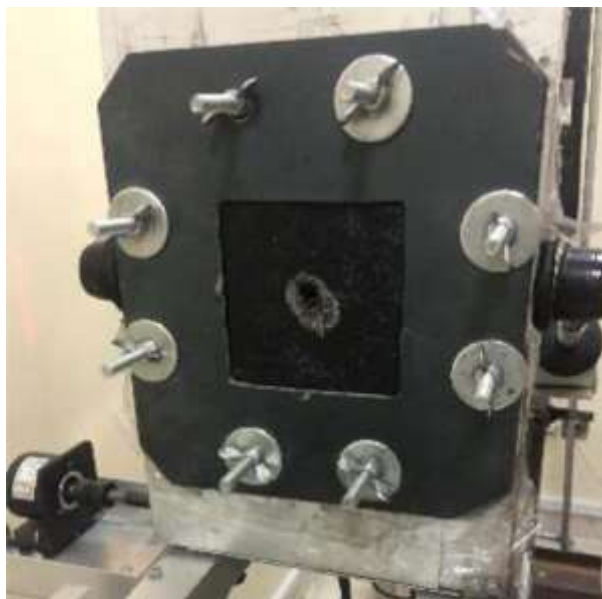


Figure 2. Composite sample in the fixture of high velocity impact test.

2.5. Characterization

For confirmation of the surface modification of GNPs, Fourier transform infrared (FTIR) spectroscopy (JASCO, 460-plus) in the range of $400\text{--}4000\text{ cm}^{-1}$ with sensitivity of 4 cm^{-1} was used. To examine the fracture surfaces, field emission scanning electron microscopy (FESEM- Hitachi S4160) at voltage of 15 kV was employed.

3. Results and Discussion

3.1. FTIR Analysis of Surface Modified GNPs

The FTIR spectra for the pristine and surface modified GNPs are depicted in Figure 3. For the pristine GNPs (Figure 3(a)), the peak at wavenumber of 3383.4 cm^{-1} was pertinent to the vibration of the hydroxyl (OH) groups. The observed peaks at 1174.3 and 2875.6 cm^{-1} were pertinent to the formation of C=O and C-H bands, respectively. However, for the surface modified GNPs (Figure 3(b)), two new peaks at wavenumbers of 983.7 and 1025.4 cm^{-1} appeared as a result of bond formation between Si-O-Si and Si-O-C, respectively. The

emergence of these two peaks endorsed the surface modification treatment of GNPs with silane coupling agent [26,27].

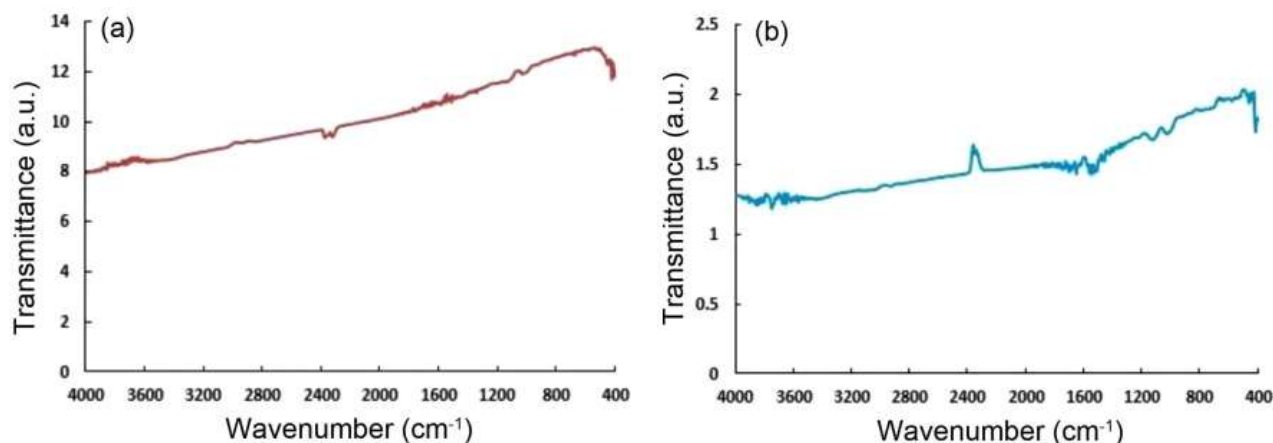


Figure 3. FTIR spectrum of (a) pristine GNPs and (b) silanized GNPs.

3.2. Results of High Velocity Impact Test

Results from the impact tests are presented in Table 2 and graphically shown in Figures 4 and 5. The maximum improvement for both impact limit velocity (11 %) and energy absorption (23 %) were achieved for the sample containing 0.3 wt.% GNPs. The lower values achieved for specimens containing 0.4 and 0.5 wt.% GNPs were attributed to the degradation of the fibers-matrix interface due to the formation of GNPs agglomerates. The agglomerates acted as stress concentration sites from which cracks easily initiated causing degradation of properties [28]. Therefore, 0.3 wt.% GNPs was the optimal amount for maximum improvement. This enhancement may be justified mainly due to 3 factors. One factor is that the frictional slippage at the fiber-matrix interface against the applied load is hindered. This is further enhanced by the mobility of polymer chains being confined due to the presence of GNPs. Therefore, the applied load can be more effectively transferred from the matrix to the

reinforcing fibers [29,30]. The second factor may be related to the higher stiffness of the GNPs compared to the epoxy matrix [31-33].

And the third factor attributed to the GNPs acting as a toughening agent. The interfacial area between epoxy and GNPs has different properties from the matrix and nanofiller. That area due to molecular interactions between the matrix and the GNPs, might contribute to energy absorption and toughening. On the other hand, due to presence of GNPs, the fracture toughness of composite can be improved by crack deflection mechanism. Moreover the surface of graphene is wavy. The wavy surface of GNPs leads to mechanical interaction between graphene and the polymeric chain and result in an increase in fracture toughness [34].

Table 2. Results of high velocity impact tests

GNPs content (wt.%)	Initial velocity (m/s)	Residual velocity(m/s)	Impact limit velocity (m/s)	Energy absorption (J)	Damage area (cm ²)
0	116	85	78.93	84.11	19.23
0.1	116	81	83.03	93.08	21.98
0.2	116	78	85.86	99.52	24.72
0.3	116	76	87.63	103.68	26.49
0.4	116	80	84	95.25	21.56
0.5	116	83	81.03	88.65	17.65

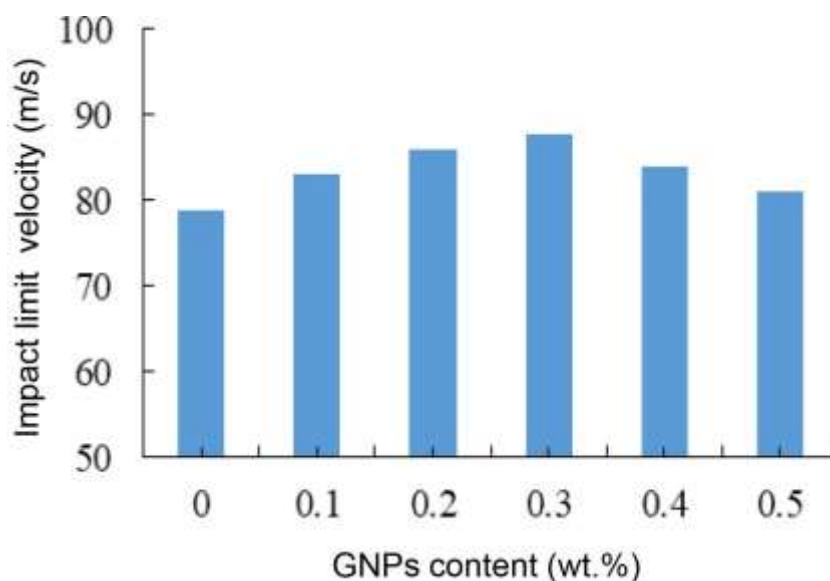


Figure 4. Impact limit velocity vs. %GNPs.

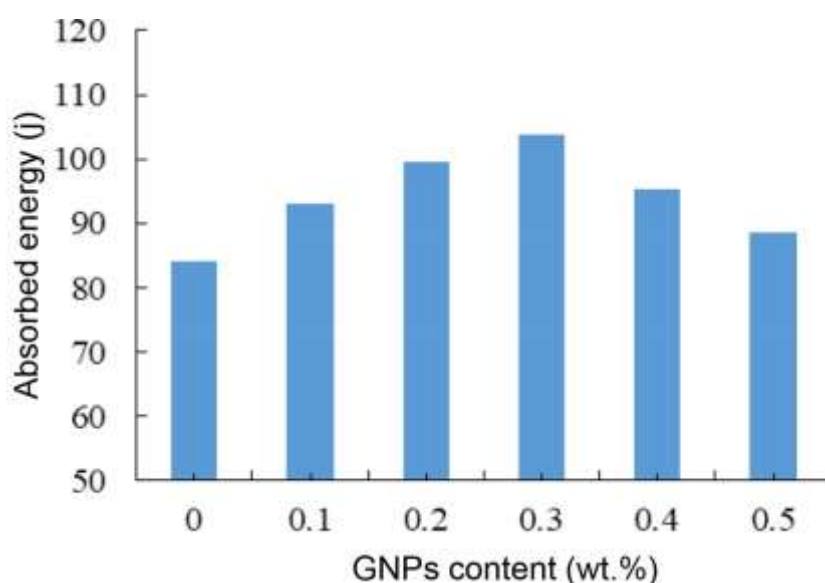


Figure 5. Absorbed energy vs. %GNPs.

3.3. Evaluation of Damage Patterns

In the high velocity impact test, the larger the damaged area, the larger the energy absorption, too. Every high velocity impact event including two stages: dentation and penetration. Compressive strength in the first stage and flexural strength in the second one can

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affect the impact properties of the laminated composites. The fundamental energy absorption mechanisms upon impact of the cone-shaped projectile onto the composite are fiber breakage, delamination, matrix cracking, shear plugging, friction between the composite and the projectile and cone formation on the back of the composite [35-37]. As observed in Table 2 and Figure 6, the damaged areas became larger by the addition of GNPs, and maximized at 0.3 wt.% GNPs, indicating the highest energy absorption capability amongst the composites. On the other hand, for the higher wt.% GNPs, e.g., 0.5 wt.%, smaller damaged areas implied lower absorbed energy, which is attributed to the agglomeration of GNPs at higher weight percentages.

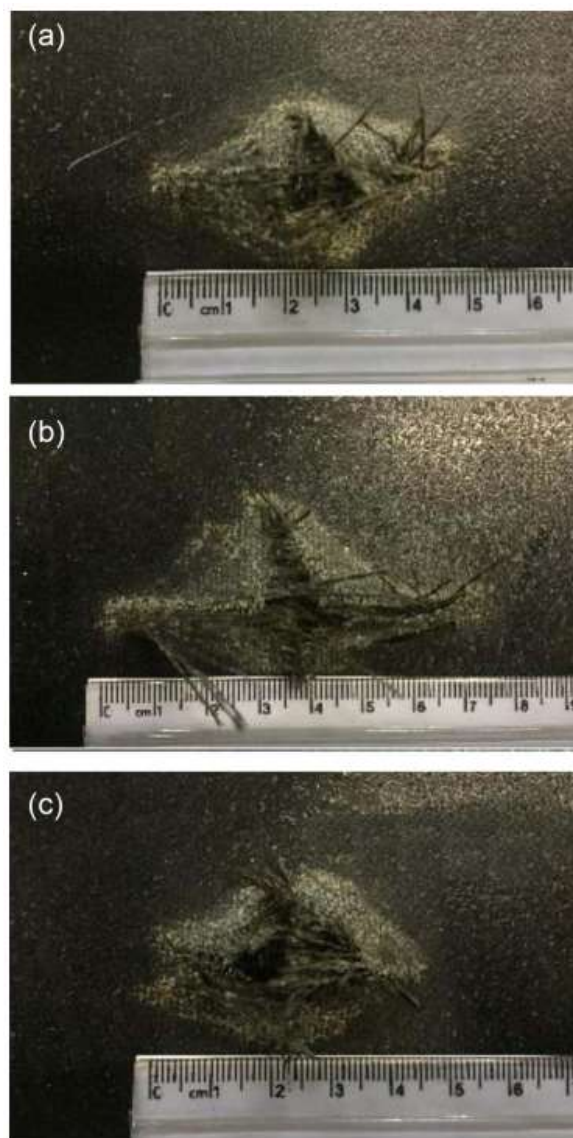


Figure 6. Damage area of composites (a) without GNPs, (b) with 0.3 wt.% GNPs, and (c) with 0.5 wt.% GNPs.

3.4. Fracture Surface Observations

The FESEM images from the fracture surfaces of the composites are shown in Figure 7. For the one without GNPs (Figure 7(a)), the matrix is wholly detached from the fibers and the smooth surface of the basalt fibers indicates weak adhesion between the matrix and basalt fibers. On the other hand, for the 0.3 wt.% GNPs composite (Figure 7(b)), the matrix is almost fully adhered to the fibers, clear indication of strong adhesion at the interface.

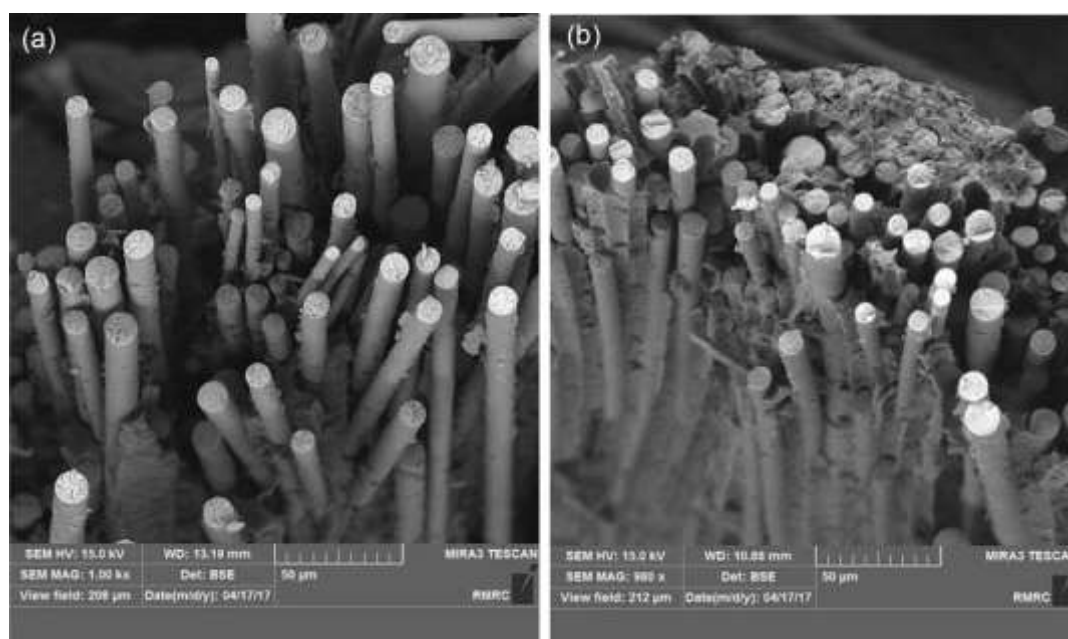


Figure 7. Fracture surfaces of basalt fibers/epoxy composites (a) without GNPs and (b) with 0.3 wt.% GNPs.

The FESEM images focused on the epoxy matrix of the composites are presented in Figure 8. For the matrix without GNPs (Figure 8(a)), the fracture surface is flat and smooth, indicating brittle fracture. In contrast, the matrix containing 0.3 wt.% GNPs (Figure 8(b)) has a rough surface and crack deflections from around the GNPs are clearly observable. In the sample containing 0.5 wt.% GNPs, however, the formation of GNPs agglomerates in the matrix is quite obvious as shown in Figure 9. As mentioned earlier, this agglomeration of GNPs caused stress concentration sites, which resulted in the loss of the mechanical properties.

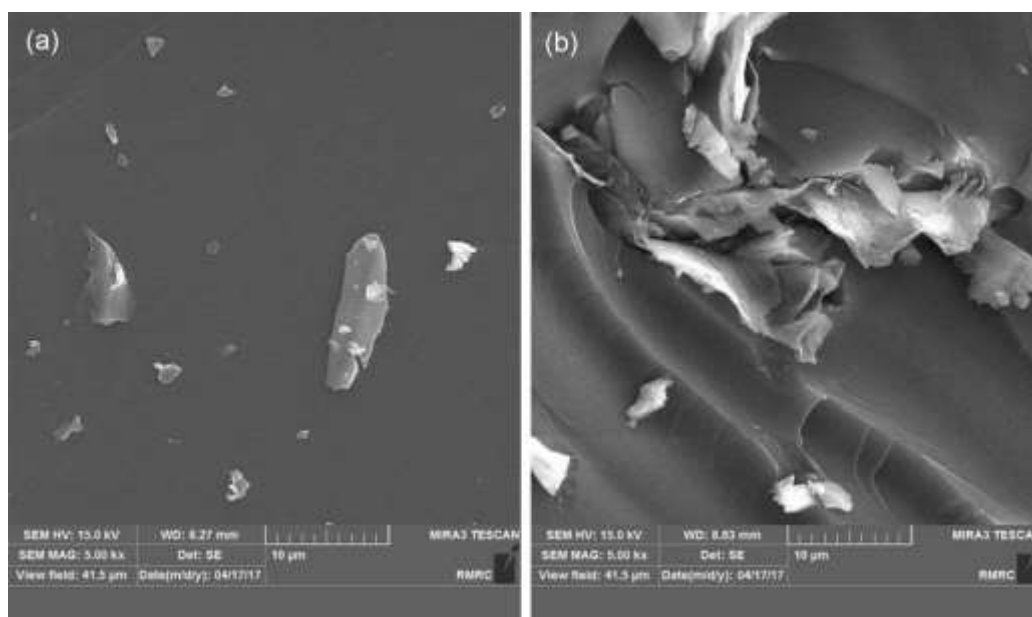


Figure 8. Fracture surfaces of basalt fibers/epoxy composites (a) without GNPs and (b) with 0.3 wt.% GNPs.

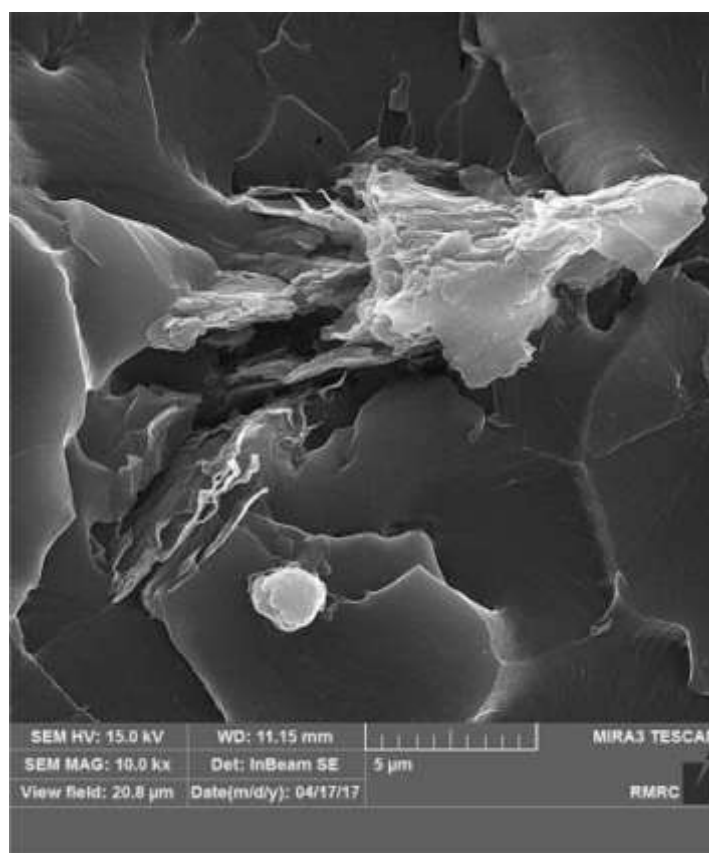


Figure 9. Agglomeration of GNPs in specimens with 0.5 wt.% GNPs.

4. Conclusion

The high velocity impact response of basalt fibers/epoxy composites containing various amounts of GNPs dispersed in the matrix was studied. The following conclusions were drawn:

1. The optimum amount of GNPs for maximum enhancement was 0.3 wt.%.
2. The impact limit velocity and energy absorption of the 0.3 wt.% GNPs nanocomposite improved by 11 and 23 %, respectively.
3. At higher weight percentages (0.4 and 0.5), the agglomeration of GNPs in the epoxy matrix caused lowering of the impact performance.
4. The performance enhancement by the addition of GNPs is attributed to stronger interfacial bond, and thus a more efficient stress transfer from the epoxy matrix to the basalt fibers.
5. The maximum damage area from the impact test occurred in the 0.3 wt.% GNPs nanocomposite, which justified the highest energy absorption capability amongst the samples.

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