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The Flexural and Impact Behavior of the Laminated Aluminum-Epoxy/Basalt Fibers Composites Containing Nanoclay: An Experimental Investigation

Farid Bahari-Sambran [1], Reza Eslami-Farsani [1]*, Shabnam Arbab Chirani [2]

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

[2] ENI Brest, FRE CNRS 3744, IRDL, Brest, France

* Corresponding author: eslami@kntu.ac.ir

Abstract

In this study, the effect of different weight percents of modified/ unmodified nanoclay particles on the flexural and impact properties of fiber metal laminates (FMLs) made of 2024-T3 aluminum sheets and basalt fibers as the reinforcements and epoxy as the matrix was investigated. As a first step, the surface of nanoclay particles was modified by the silane coupling agent. The creation of the functional groups on the surface of nanoclay particles was confirmed by Fourier transform infrared spectroscopy (FT-IR) analyses. The modified nanoclay with different weight percents of 0, 1, 3 and 5 was added into the epoxy matrix; then, for the better distribution and dispersion of nanoparticles in the matrix, the mechanical and ultra-sonication machines were used. Also, to ensure better interaction and adherence between the matrix and the aluminum sheets, the mechanical and chemical treatments were conducted. Then, the mixture of epoxy and nanoclay with woven basalt fibers and aluminum sheets was used to fabricate FMLs. To survey the effect of these nanoparticles on the mechanical properties, the 3-point bending test and the high velocity impact test were used. The results showed that the maximum effect of adding nanoclay particles on the flexural and impact behavior was obtained by using 3 wt.% of the modified nanoclay. These properties of FMLs, which contained the 3 wt.% unmodified nanoclay, were weakened in comparison to a similar specimen containing the modified nanoclay. Furthermore, the flexural modulus was enhanced by increasing the weight percent of the nanoparticles. Also, to investigate the fracture mechanism, the field emission scanning electron microscope (FESEM) was used. The microscopic images revealed that adding nanoclay particles led to the improvement of the interaction between the matrix and basalt fibers, thereby improving the flexural and impact properties.

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Keywords: Fiber metal laminates, Nanoclay, Surface modification, Flexural properties, Impact properties

1. Introduction

Composite materials have received much attention in recent years. They have an extraordinary advantage over the conventional metallic alloys when it comes to the specific strength. Fiber-metal laminates (FMLs), categorized as hybrid composite materials, are made from thin metallic layers and fiber reinforced polymers. The fiber reinforced polymeric layer could act as a core between two adjacent metallic laminates [1, 2]. FMLs show such enhanced properties as resistance against fatigue, strength, fracture toughness, resistance against impacts, and a high capacity in energy absorbing [2].

The initial goal in producing FMLs is to combine the high fatigue resistance of composite materials with the stability and plastic behavior of metallic laminates. Furthermore, the need for improved strength in fatigue resulting from cyclic loadings, especially in aluminum alloys, is another reason for their development. The interface between different layers in FMLs results in the effective energy dissipation and bridging behavior of fibers, hindering crack propagation; hence, compared to the monolithic materials with the same thickness, improved resistance to cyclic stresses could be obtained [1, 3].

The early researches focused on FMLs due to their suitable fatigue resistance; however, it turned out that the interface of metal and composite layers could play a great role in the impact resistance of FML composites. Previous studies have shown that FMLs have less extended internal damage when compared to the conventional composites; they also have a considerably higher resistance to destruction, as compared i to monolithic metallic materials [4, 5]. The main application of FMLs is in the airplane industry, where it is necessary to

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analyze the destruction caused by low-velocity and high-velocity impacts. The former might happen due to the dropping of a tool during the maintenance services, while the latter might occur in take-off, when small flakes bump into the airplane or in the case of hailing [5].

It should be noted that glass, carbon and Kevlar fibers are among the most common reinforcing fibers in the epoxy matrix. However, basalt fibers, which are known as a natural (inorganic) fiber, due to their unique properties, as compared with the common ones, have attracted more attention. High strength, excellent adhesion to a resin, sound insulation, low water absorption, high resistance against chemical corrosion, no environmental risks, high temperature functioning range, and low production cost in comparison to carbon fibers are some of their special features [6]. They also have higher tension strength, fracture strain, and more heat resistance, in comparison to glass and carbon fibers; therefore, they can replace glass fiber reinforced composites in many applications such as aerospace, transportation, and automobile industry [7-10]. Basalt fibers were utilized for the first time for FMLs fabrication under impact loading conditions by Eslami-Farsani et al [11].

Regarding the ongoing research in nanotechnology, polymer-based nanocomposites have been of much interest. The discovery of laminated clays, which could significantly enhance the mechanical properties through a systematic modification of polymers, was a breakthrough in this field. Clay, considered as a Montmorillonite, is made of small layers or an internal octahedral layer located in the middle of two tetrahedral silicate layers. An aluminum oxide laminate in which some atoms have been substituted with magnesium atoms might be considered as an octahedral layer. Clay laminates are naturally available in a cluster whose interactions with polymer chains could be categorized in immiscible, intercalated, and exfoliated structures. Based on the studies previously done, in the immiscible form, the polymer chains cannot penetrate into the interlayer spaces, whereas in the intercalated

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structures, the polymer chains get the opportunity to infiltrate in the layers. It is worth noting that in the exfoliated state, the polymer chain can separate the interlayer spaces and completely distribute theses layers in the polymers. If the nanoclay is distributed uniformly in the polymeric matrix, or, to put it in another way, if it is misciible, the desired properties would be attained to a large extent. The main reason for the addition of fillers to the polymers is the enhancement of their module or stiffness through employing reinforcing mechanisms or utilizing composite-related theories. A plane distribution of random nanoclay laminates is highly effective in improving the stiffness [12].

Bashar et al [13] investigated the effect of adding the modified nanoclay with various weight percents (0, 1, 2, 3 and 4) on the mechanical properties of epoxy based composites. Their study revealed that the addition of nanoclay up to 3 wt.% could result in 20% improvement in the stiffness. However, nanoclay might cause a negligible loss in the tension strength of the nanocomposite. They have shown that this addition could have no side effects on the fracture energy, since the main resistance mechanism against crack propagation is the deflection of it, which leads to a slight energy loss.

Subagia et al. [7] studied the effect of Tourmaline nanoparticles addition with various weight percents (0, 0.5, 1, 1.5 and 2) on the flexural properties of the reinforced composites by basalt fibers, showing that the maximum improvement in these properties would result from 1 wt.% Tourmaline. This would cause a 16% increase in the bending strength and a 153% rise in the bending module.

Ho et al [14] investigated the effect of nanoclay addition with various weight percents ranging from 0 to 8 percent, as was performed through mechanical mixing, on the mechanical behavior of epoxy-based composites. The results showed the best optimized tensile strength and stiffness in 5 wt.% of nanoclay.

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Ferrante et al. [5] presented their research on the impact behavior of three different laminated composites made of Basalt fibers, aluminum, and epoxy resin. The results revealed the positive effect of basalt fibers on the improvement of the impact behavior in FMLs, as compared to the glass fibers.

Rostamian et al. [15] experimentally investigated the effect of nanoclay and Silica nanoparticles on the flexural strength of epoxy-glass fibers composites. With the 4 wt.% modified nanoclay and 3.5 wt.% of silica nanoparticles, flexural strength was enhanced by 11 and 63 percent, respectively.

Poll et al. [16] also studied the tensile properties in multi-layered glass fibers-epoxy composites consisting of woven fibers. Nanoclay was added on a 0, 3, 5, 7, and 10 wt.% basis to the matrix, showing a negative influence on the mechanical properties of nanocomposites in the lower weight percent. Moreover, the most energy absorbing happened upon 5 wt.% addition of the nanoclay.

Caprino et al. [17] also investigated the effect of impact velocity, weight, and energy on the laminated basalt fibers-aluminum composites produced from aluminum 2023-T3 sheets and epoxy. They showed that the force-displacement diagram was generally affected only by the energy level of the impact and was independent of the speed and weight of the projectile.

Vlot [18] further compared the static loading of projectile, high-velocity impact, and low- velocity impact on various FMLs (ARALL and GLARE) made from the aluminum sheets of 7975-T6 and 2024-T3. The results revealed a correlation between the dent depth of the impact and the type of aluminum alloy used in the study.

Yaghoubi et al. [19] also studied the reactions of FMLs with a GLARE core against low-velocity impact, by considering such variables as projectile's weight and sample thickness. They showed that the energy threshold of the crack would change parabolically and

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perforation might happen sooner due to a reduction in the thickness and an increase in the projectile's weight.

Poll et al. [20] also investigated the ballistic behavior of woven glass fibers-epoxy nanocomposites containing the modified nanoclay with various weight percents of 0, 3, 5, 7, and 10. The impact tests of 134 and 169 m/s indicated the most energy absorbing at 134 m/s and 169 m/s with 3 and 10 wt.% of nanoclay, respectively.

In this study, according to the survey of the literature, the effects of the modified nanoclay on the flexural and impact properties of FMLs (aluminum 2024-T3-epoxy/basalt fibers), as compared to the control sample (without nonoclay), were investigated. Furthermore, the effect of the modification of nanoclay on the flexural and impact properties of samples was studied

2. Experiment

2.1. Materials

2024-T3 aluminum sheets with 0.5 mm thickness were used in this study. In the production procedure of the composite samples, the epoxy resin of Epon 828 along with a polyamine hardener (from mokarar) was utilized as the matrix. Furthermore, woven basalt fibers (from Basaltex) and nanoclay (Montmorillonite k10 from Sigma-Aldrich) were used as the reinforcement. Suitable adhesion between aluminum sheets and the polymer matrix was achieved by using sodium-hydroxide solution as the etchant. Finally, the organo-silane tri-glycidoxy propyl trimethoxysilane (3-GPTS) coupling agent (from Merck) was utilized for the proper distribution and interaction between polymer chains and clay laminates.

2.2. Preparation and surface treatment of aluminum sheets

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2024-T3 aluminum sheets were cut with the dimensions of 25×150 mm^2 and 130×130 mm^2 to develop flexural and impact samples, respectively. The shape of samples is schematically illustrated in Fig. 1. Subsequently, mechanical and chemical treatments were conducted on them in order to establish the proper adhesion among the aluminum sheets and the polymer matrix. The mechanical treatment was performed through sanding with a 100˚ 45-angle sander. The samples were put in a 0.5% sodium hydroxide solution at 60 ˚C for 3 min., afterwards, this was done to ensure chemical corrosion and the necessary adhesion in the interface of aluminum sheets with the polymer matrix.

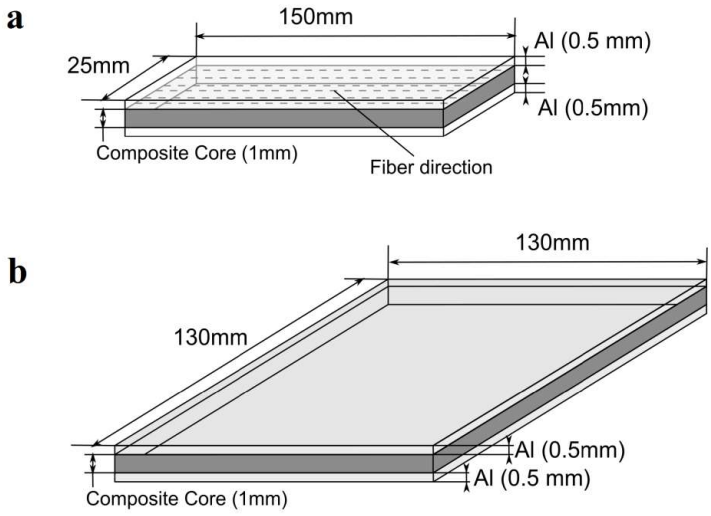

Fig. 1. Schematic of FML samples for (a) the flexural test, and (b) the high velocity impact test.

2.3. Surface modification of nanoclay

In order to functionalize nanoclays, they were put into a solution of ethanol and distilled water and distributed by the use of an Ultrasonic Probe device. Silane agent (3-GPTS) was added at this stage. The resultant solution was subsequently treated in a reflux system of refining for 12 hours at 70 ˚C. Finally, to separate the surface modified nanoclay from the

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solution, it was centrifuged at 4000 rpm. Also, for segregating the non-functioned silane compounds or nanoclay, the obtained substance was fully washed with ethanol and dried completely in an oven. To confirm the modification of the nanoclay, Fourier transform infrared spectroscopy (FT-IR) analysis (Jasco-460 plus device) was performed.

2.4. Fabrication of FMLs

For the preparation of the matrix, surface-modified nanoclays were initially added to the resin (at 1, 3, and 5 wt.%) and later mixed by means of a mechanical mixer for 20 min and at 2000 rpm. In order to gain an adequate distribution of the surface- modified nanoclay in the matrix and break the clusters of nanoclay down into smaller ones, the mixture was subjected to a 60 min. exposure with ultrasonic waves via a Homogenizer probe (from Fapen Co.). Four layers of woven basalt fibers with a 50-50 weight ratio were used in the production of the composite samples. Meanwhile, a 10 wt.% hardener was prepared and added to the mixture,

The fabrication was completed by the hand layup method. For the sake of comparison, some samples were made similarly, excluding the nanoclay in the procedure.

A fixture was utilized to clamp the samples prior to curing and apply a uniform pressure to them. The samples were pressed for 24 hr at 5 bar at room temperature. To achieve the maximum strength and a full-cure treatment, the flexural and impact tests were performed after 20 days.

2.5. Flexural test

A three-point support flexural test was conducted on the FMLs samples using the D790-17 ASTM standard and a 10-ton Koopa machine, and the 150×25 mm^2 samples were mounted on two support spans [21]. Moreover, the three-point flexural test was carried out

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with at least five samples and the average of results were reported. Based on the ASTM standard, the distance of the supports was considered to be 32 times of the sample's thickness. The samples were then loaded with a constant strain according to Eq. (1), and the bending force-displacement diagram was derived and drawn by the machine. The results were used in Eqs. (2) and (3) to calculate the flexural stress and strain [21, 22].

$$R = ZL^2/6d \tag{1}$$

$$\delta_f = 3PL/2bd^2 \tag{2}$$

$$\varepsilon_f = 6Dd/\pi L^2 \tag{3}$$

where R is the rate of crosshead motion in mm/min, Z is the straining rate of the outer fiber in mm/mm/mm, which is equal to 0.01, P is the loading in N, L is the distance of the two supports in mm, d is the thickness of the FML samples in mm, b is the width of the bending sample, and D is the midspan deflection in mm.

2.6. High-velocity impact test

The high-velocity impact test was performed on the FMLs samples by means of a gas gun (model M211) which could measure the residual velocity (Fig. 2). The 130×130 mm^2 samples were clamped by a square fixture. For each nanoclay, content five samples were tested. The velocity of the impact was set to be 118 m/s and the conical projectile was made from some commercial aluminum with the length of 40 mm, the diameter of 21 mm, and the weight of 27 g.

Based on Eqs. (4) and (5), the results were used for the calculation of limit velocity and the absorbed energy.

$$E_{50} = \sqrt{V_i^2 - V_r^2} \tag{4}$$

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$$E_{abs} = 1/2(V_i^2 - V_r^2) \quad (5)$$

where V_{50} is the limit velocity, E_{abs} is the absorbed energy, V_I is the input velocity, and V_r is the residual velocity.

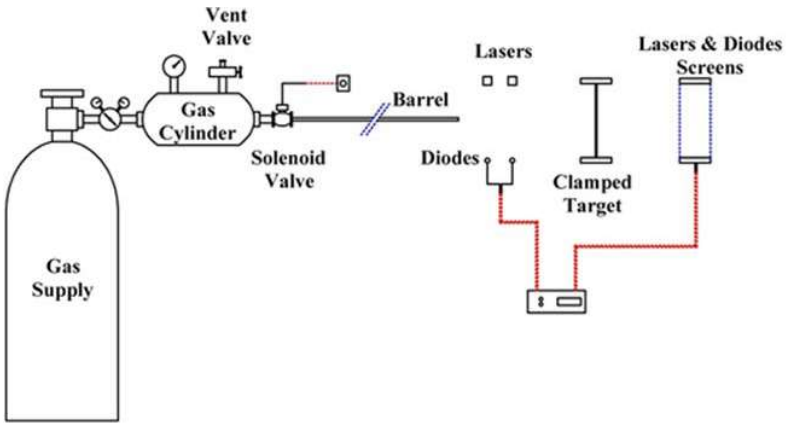

Fig. 2. Schematic of the gas gun [23].

2.7. Study of the fracture surface and debonding area of FMLs

To investigate the debonding and the fracture surface of the tested samples and define the active mechanisms showing the influence nanoclay on the flexural and impact behavior of FMLs, microstructural studies were conducted. Therefore, a MIRA3TESCAN-XMU model of field emission scanning electron microscopy (FESEM) was used.

3. Results and Discussion

3.1. FT-IR spectroscopy

FT-IR diagram of the initial nanoclay as well as the surface- modified ones is shown in Fig. 3. A wide peak could be seen in the FT-IR spectrum of the surface- modified nanoclay at 3402 cm^{-1}, indicating the tensile vibration of the hydroxyl group. The peak that happened at 1614 cm^{-1} also implied the bending vibration of H-O-H [24]. Tensile vibration corresponded to hydroxyl groups attached to aluminum atoms which could not be found in the initial

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nanoclay spectrum, as illustrated in the band related to 3620 cm^{-1} as well [25] . The absorbing that occurred at 1029 cm^{-1} was also due to some tensile vibration in Si-O and Si-O-Si [24].

Furthermore, the absorption at 456 and 530 cm^{-1} peaks defined a bending vibration in the Si-O-Al and Si-O-Si groups of the surface- modified nanoclay [26]. The peak of 1215 cm^{-1} also denoted the vibrating of the epoxide ring. This could not be seen in the unmodified nanoclay spectrum, suggesting a 3-GPTS bond in such nanoclays.

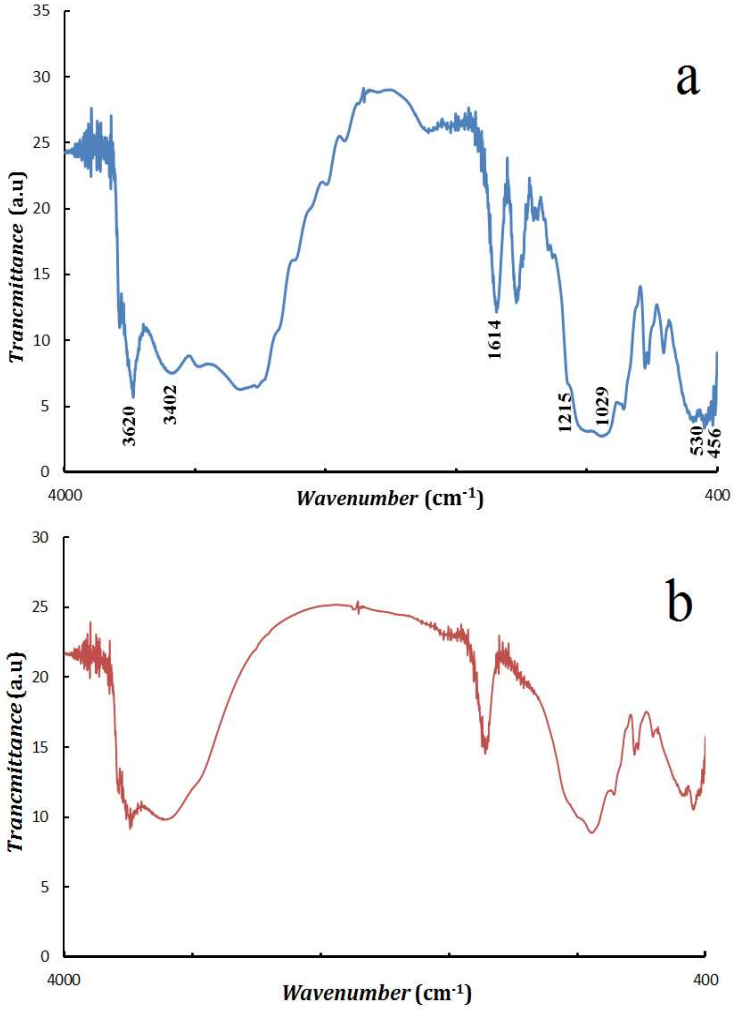

Fig. 3. FT-IR spectra of (a) nanoclay/ 3-GPTS, and (b) nanoclay.

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3.2. The interaction between the aluminum sheet and the matrix

The grooves as well as the flaws and surface roughness caused by mechanical and chemical treatments, respectively, resulted in the further engagement of the matrix and aluminum sheets (Fig. 4). The addition of the nanoclay, on the other hand, led to a rise of the viscosity [27, 28]. This was not very tangible during the mixing and homogenization stages. The effect of the viscosity rise could be observed in two aspects of FMLs. First, the number of air pockets trapped in the matrix after the cure was affected by the increase in viscosity, leaving no choice except extracting all of them due to the high viscosity of the mixture. The resultant vacancies would weaken the overall mechanical properties of the composite. Second, the viscosity increase resulting from the addition of nanoclay would change the wettability and liquidity characteristics of resin on the aluminum surface, thereby reducing the quality of adhesion [29]. However, due to their high inherent modulus, nanoclay would cause a rise in the matrix modulus, making the exit of resin from the grooves and flaws created by the treatment procedures much harder.

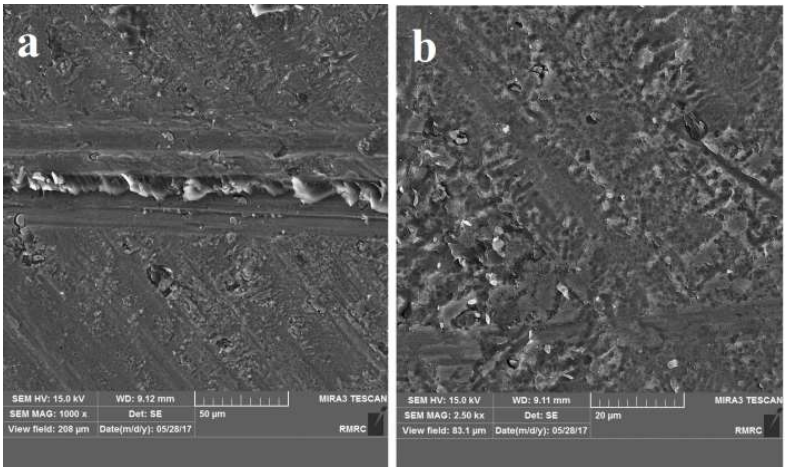

Fig. 4. Engagement between the aluminum sheet and epoxy after curing (a) engagement in grooves created by mechanical treatment, and (b) engagement and penetration in the surface roughness created by chemical treatment.

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3.3. Results of the flexural test

The results of the three-point flexural test on FMLs consisting of various weight percents of nanoclay are illustrated in Table 1. Since the most efficiency would be obtained at 3 wt.% of nanoclay, for the sake of comparison, a similar 3 wt.% sample containing the unmodified nanoclay was made and tested in the same way.

Table 1. Results of three point flexural test of FML specimens

Nanoclay loading (wt.%)	Flexural strain (%)	Flexural strength (MPa)	Flexural modulus (GPa)	Energy absorption (J/mm^3)
0	1.30±0.04	502.87±7.51	50.59±1.90	3.26±0.05
1	1.68±0.06	568.10±17.44	74.30±2.75	4.74±0.14
3	2.85±0.05	766.94±14.71	99.56±2.41	9.64±0.21
5	1.52±0.04	641.30±11.56	122.22±2.55	4.87±0.10
3 um*	1.47±0.05	583.31±12.36	94.86±2.07	4.44±0.09

* um (unmodifiied nanoclay)

The effect of nanoclay addition on the flexural strength of FML samples is illustrated in Fig. 5. As can be seen, the variations in the flexural strength of the samples were initially raised with the increase in the nanoclay weight percent and then decreased. The maximum flexural strength was achieved in 3 wt.% of the modified nanoclay. A 52% enhancement from 502.87 MPa up to 766.94 MPa was obtained in the flexural strength of such samples, as compared to the control sample. The boost came from the improvement made in the interface characteristics of the epoxy matrix and basalt fibers. This would facilitate the transfer of

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loading from the matrix to the fibers; due to the high strength of basalt fibers in comparison to the matrix, it would result in an increase in the flexural strength of epoxy based composites. On the other hand, the addition of nanoclay could lead to a rise in the modulus of the polymer matrix as well. This happened for the higher intrinsic modulus of nanoclay, as compared to the epoxy matrix, finally improving the modulus of the matrix including nanoclay. Moreover, the engagement of the polymer chains and nanoclay laminates could also give rise to the modulus. The rise in the modulus and the stiffness of the matrix would result in the reduction of the shearing stress in the interface of the aluminum layer and the fiber-reinforced composite, causing an overall increase in the flexural strength of FMLs.

The increase of nanoclay (up to 5 wt.%) would result in a 16.4% reduction from 766.94 MPa to 641.3 MPa of flexural strength, as compared with the sample having 3 wt.% of the modified nanoclay. This could be due to the decrease in the adhesion of the epoxy matrix and basalt fibers, resulting from the formation of the discrete clusters of the nanoclay in the matrix. Furthermore, these clusters would act as stress concentration points and reduce the bending strength [30]. As illustrated in Fig. 5, the flexural strength of a unmodified 3 wt.% nanoclay sample fell behind the modified one by 24%, from 766.94 MPa to 583.31 MPa. This could also be attributed to the agglomeration of the unmodified nanoclay in the matrix.

The effect of adding nanoclays on the flexural energy absorption of laminated composites is shown in Fig. 6. As shown, by increasing the amount of modified nanoclay, up to 3 wt.%, the energy required for fracture was increased and then decreased. The fracture energy of the laminated composite was raised by 46% and 195 % with the increase of 1 and 3 wt.% of the modified nanoclay, respectively, as compared to the control sample, reaching to 4.74 and 9.64 (J/mm^3). This increased fracture energy in 5 wt.% of modified nanoclay reached to 49%, as compared to the control sample. The reason for the increase of fracture

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energy with the addition of clay nanoparticles can be the improvement of the properties of the interface between the epoxy/ basalt fiber and the composite core/ aluminum sheet, which prevented the crack propagation in the interface. In addition, the mechanism of crack deflection by nanoclay increased the fracture energy. Therefore, it can be concluded that energy absorption capacity of the epoxy containing the clay nanoparticles was higher than that the neat epoxy. But the factor reducing the fracture energy in a sample containing 5 wt.% of modified nanoclay and 3 wt.% of unmodified nanoclay, as compared to the sample containing 3 wt.% of modified nanoclay, was inappropriate distribution and agglomeration of nanoclays, leading to the weakening of the interface properties and the crack deflection mechanism [31].

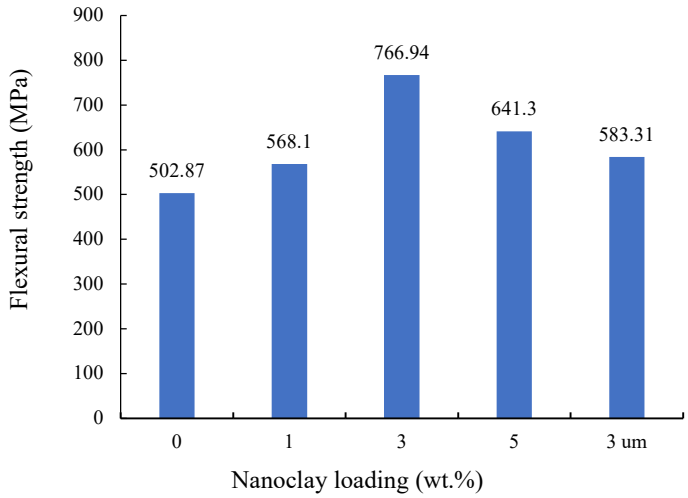

Fig. 5. Flexural strength of FMLs at different nanoclay loadings.

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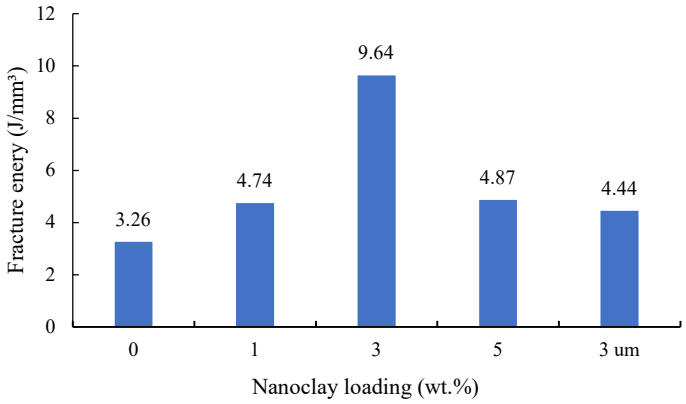

Fig. 6. Energy absorption of FMLs at different nanoclay loadings.

The results of the addition of various nanoclay percentages on the flexural modulus of FML samples have been presented in Fig. 7. As can be seen in Fig. 5, the increase in the modified nanoclay with 1, 3, and 5 wt.% would lead to an enhancement in the flexural modulus of laminated composites, as compared with the control sample, by 46, 96 and 141 percent, respectively. It was improved for 1, 3, and 5 wt.% of the modified nanoclay up to 74.30, 99.56 and 122.22 GPa, as compared with the control sample, which was 50.59 GPa, respectively. The presence of the modified nanoclay with high intrinsic modulus, in comparison with the polymeric matrix, by forming more engagement and limitations in the movement of the polymer chains, could increase the stiffness and the modulus of the laminated composite. The minor reduction seen in the specimen containing the 3 wt.% unmodified nanoclay could also contribute to the agglomeration of the unmodified nanoclays, decreasing their efficiency in terms of stiffness [29].

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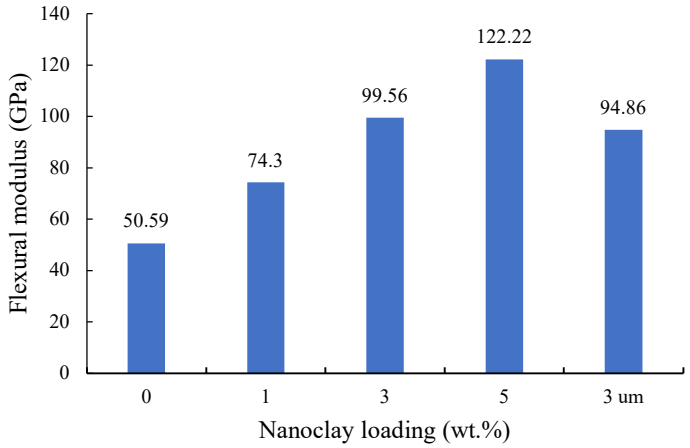

Fig. 7. Flexural modulus of FMLs at different nanoclay loadings.

3.4. Results of the high-velocity impact test

The effect of nanoclay addition on the residual velocity in the impact test has been presented in Fig. 8. As can be seen, increasing the nanoclay up to 3 wt.% could reduce the residual velocity while it went up afterwards. It had the most effect on the residual velocity at 3 wt.% of the nanoclay, which was around 63 m/s, resulting in a 10% reduction, as compared with that for the control sample, which was 70 m/s. The residual velocity of the 3 wt.% surface- modified nanoclay sample, as compared to the similar unmodified one, was declined by 12.5% from 71 m/s to 63 m/s.

Regarding the limit velocity, as can be seen in Fig. 9, it could rise up to 99.7 m/s, while by increasing the nanoclay weight percentage to 3 wt.%, there was an increase by 5 and 6 percent in comparison to the control sample and 3 wt.% of the unmodified nanoclay sample, respectively.

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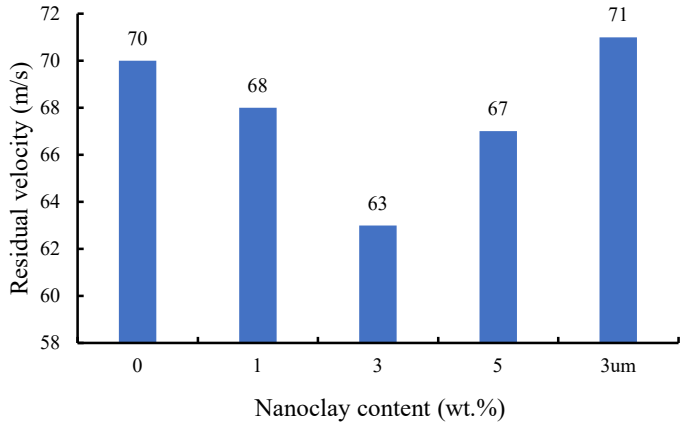

Fig. 8. Residual velocity versus the initial impact velocity for FMLs containing various nanoclay loadings.

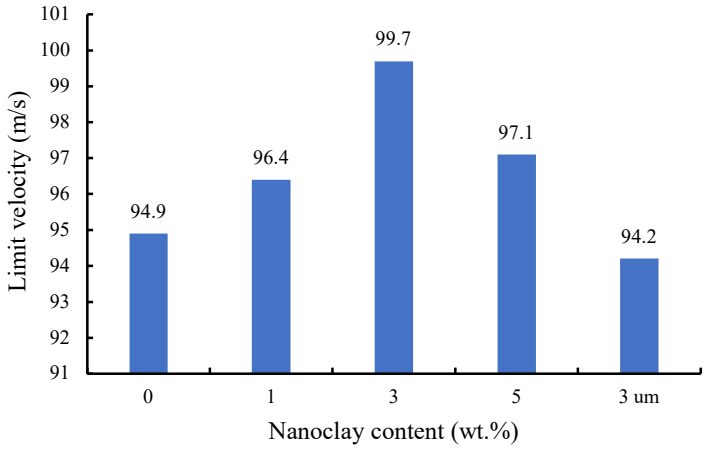

Fig. 9. Limit velocity of FMLs at various nanoclay loadings.

The effect of nanoclay addition on the energy absorption has been shown in Fig. 10. There was an upward trend up to 3 wt.% of nanoclay by 134.39 j of the absorbed energy; then it was transformed into a downward drift. Compared to the control sample and the 3 wt.% unmodified nanoclay sample, some increase of around 10 and 12% could be seen, respectively. It is noteworthy that the fracture mechanisms and energy absorption in a high-velocity impact depend not only on the projectile's velocity, weight and shape, but also on

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the mechanical properties of the FML, its combination, and the interaction between the matrix and fibers, as well as that between the matrix and the metal sheets [20]. Energy absorption mechanisms include fracture occurrence in any layer of the laminated composite, deformation of the laminated composite, delamination of the composite part, separation in the composite and metallic sections, and formation of an aluminum plug in the front section of the panel and an aluminum Petal in the back of it; for each, a different amount of energy absorption would result from any variation in the aforementioned parameters.

Nanoclays have the highest contribution to the fracture of the composite section. Therefore, elevating the energy absorption up to 3 wt.% of the surface modified nanoclay would be more promising for the improvement of the compressive strength in this section. Regarding the results of the flexural test, however, it was found that adding up to 3 wt.% of the surface modified nanoclay would increase the flexural strength, leading to an enhancement in the energy absorption. A plausible explanation for it may come from the two steps of any high velocity impact event: dentation and penetration. Compressive strength in the first step and flexural strength in the second one can affect the impact behavior of the laminated composites. Moreover, the improvement observed in the energy absorption by increasing the nanoclay up to 3 wt.% might result from the rise in the shearing strength among the layers [32, 33].

A 5% reduction in the energy absorption of the 5 wt.% surface- modified nanoclay sample, as compared with the 3 wt.% one, might come from an improper distribution of the nanoclay; consequently, their agglomeration would result in the formation of stress concentration points. Furthermore, the addition of such nanoclays to the matrix would increase resin's viscosity and imprison more bubbles in the mixture, reducing the strength.

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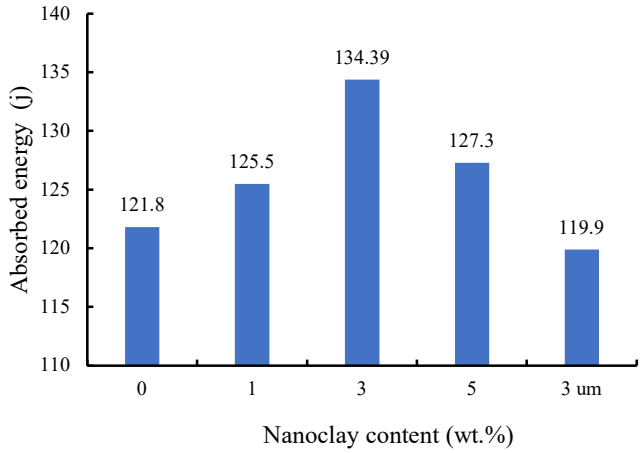

Fig. 10. Absorbed impact energy by FMLs containing various nanoclay loadings.

3.5. Investigation of the damage Area

Generally, the damage in FMLs could be categorized as a visible one including plastic indention and cracking of aluminum, and some internal damage consisting of the fibers pull out, matrix crack, delamination of the composite, and separation of the aluminum sheets from the matrix. On the other hand, a larger damage area can result in a higher amount of energy absorption. The front and back of the tested samples containing different percentages of nanoclay have been displayed in Fig. 11. Also, the destructive cross-section views of the damaged area can be seen in Fig. 12; all of the specimens were damaged completely in different modes; plastic deformation, aluminum rupture, and the fracture in both fibers and the matrix could be easily seen. Fig. 11b shows a 3 wt.% nanoclay sample damaged more thoroughly than the other samples. The delamination of the aluminum and composite interface also happened more remarkably, as compared to the others. This can easily be observed in Fig. 12b, where the internal damage of the sample in the form of a separation between the composite and the matrix has been shown; in some parts, even the delamination

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of the composite could be spotted, resulting in more energy absorption with respect to the other samples.

A 3 wt.% unmodified nanoclay sample is demonstrated in Figs. 11d and 10d; a rupture of aluminum and breakage of fibers experiencing lower damage area, as compared with the other samples, could be seen. This sample revealed the least amount of energy absorption during the impact tests.

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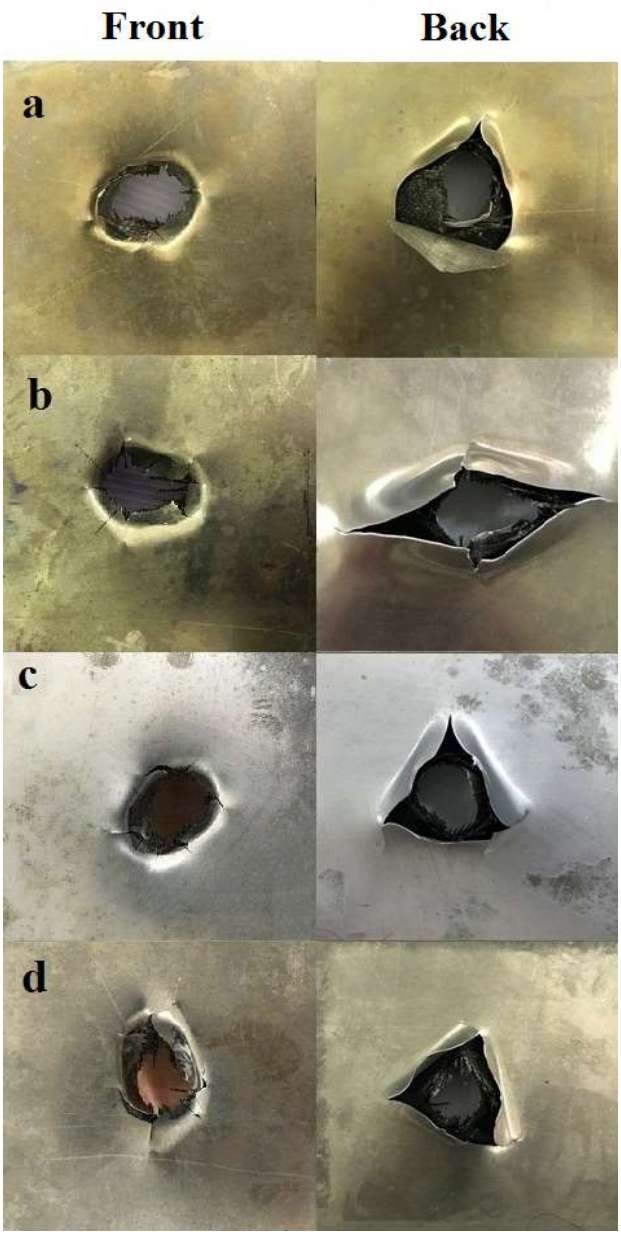

Fig. 11. Front and back images of FMLs after high velocity impact testing (a) without nanoclay, (b) with the 3 wt.% modified nanoclay, (c) with the 5 wt.% modified nanoclay, and (d) with the 3 wt.% unmodified nanoclay.

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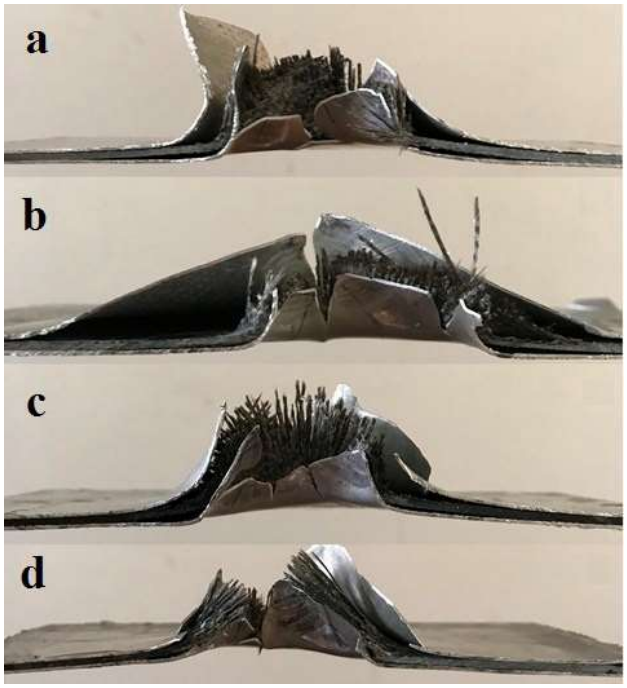

Fig. 12. Destructive section views of the impacted areas testing (a) without nanoclay, (b) with the 3 wt.% modified nanoclay, (c) with the 5 wt.% modified nanoclay, and (d) with the 3 wt.% unmodified nanoclay.

3.6. Study of the fracture surface of FMLs

The fracture region that resulted from the impact test is shown in Fig. 13. The fracture surface of the control sample could be seen in Fig. 13a. Basalt fibers had a clean and free-of-resin-adhesion surface; however, for the 3 wt.% nanoclay sample (Fig. 13b), the matrix containing the reinforced epoxy by the modified nanoclay was still stuck to the basalt fibers and not separated in most of the parts. The 3 wt.% unmodified nanoclay sample is also shown in Fig. 13c; although the adhesion of the matrix could be spotted in some places of the fibers, it was not as sticky as the modified sample. Therefore, it could be concluded that the fracture happened in the matrix for the nanoclay-free samples through the separation of the matrix and

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fibers, while in the specimens containing nanoclay, the cohesion of the matrix and the fibers verified the desired adherence between them resulting in the fracture of the fibers [34, 35].

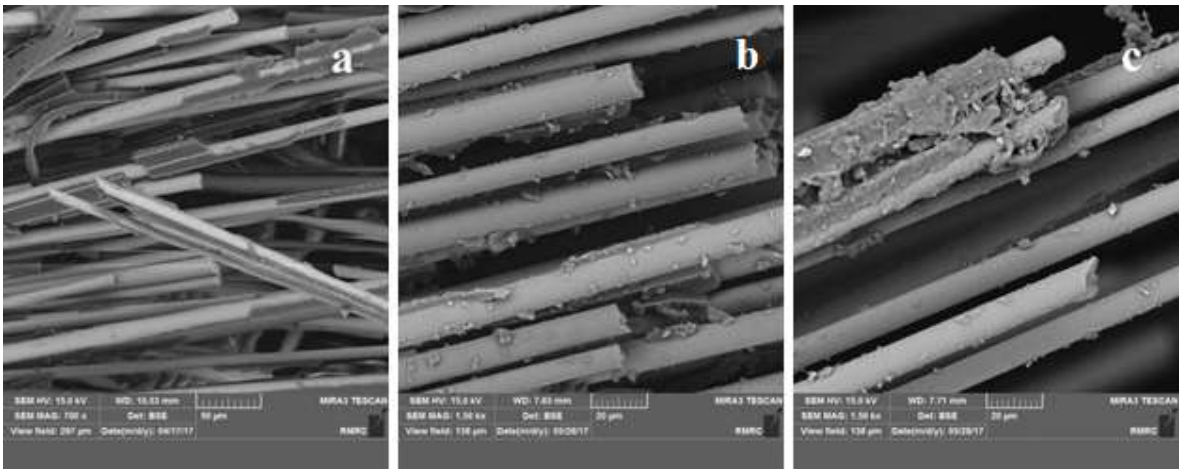

Fig. 13. FESEM micrographs of the fracture surfaces of FMLs (a) neat epoxy, (b) with the 3 wt.% modified nanoclay, and (c) with the 3 wt.% unmodified nanoclay.

FESEM images of nanoclay composites are shown in Fig. 14. Based on Fig. 14a (displaying the 3 wt.% surface- modified nanoclay sample), clay layers were distanced from each other and intercalated, showing the presence and engagement of polymer chains between the layers. Surface- modified clay laminates could be easily seen in the picture. Fig. 14b shows the fracture surface of the sample containing 5 wt.% of the surface- modified nanoclays, where a 15 micron cluster consisting of clay layers could be seen. The stress concentration in the surroundings of this cluster would result in the cracks and small-scale notches. A 3 wt.% unmodified nanoclay sample is also illustrated in Figs. 14c, and 12d with two different magnifications, where nanoclays were agglomerated rather than being distributed uniformly in the matrix. The cluster not only formed stress concentration around

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itself, but also prevented polymer chains from engaging in that region, bringing about fragility and brittleness in the area.

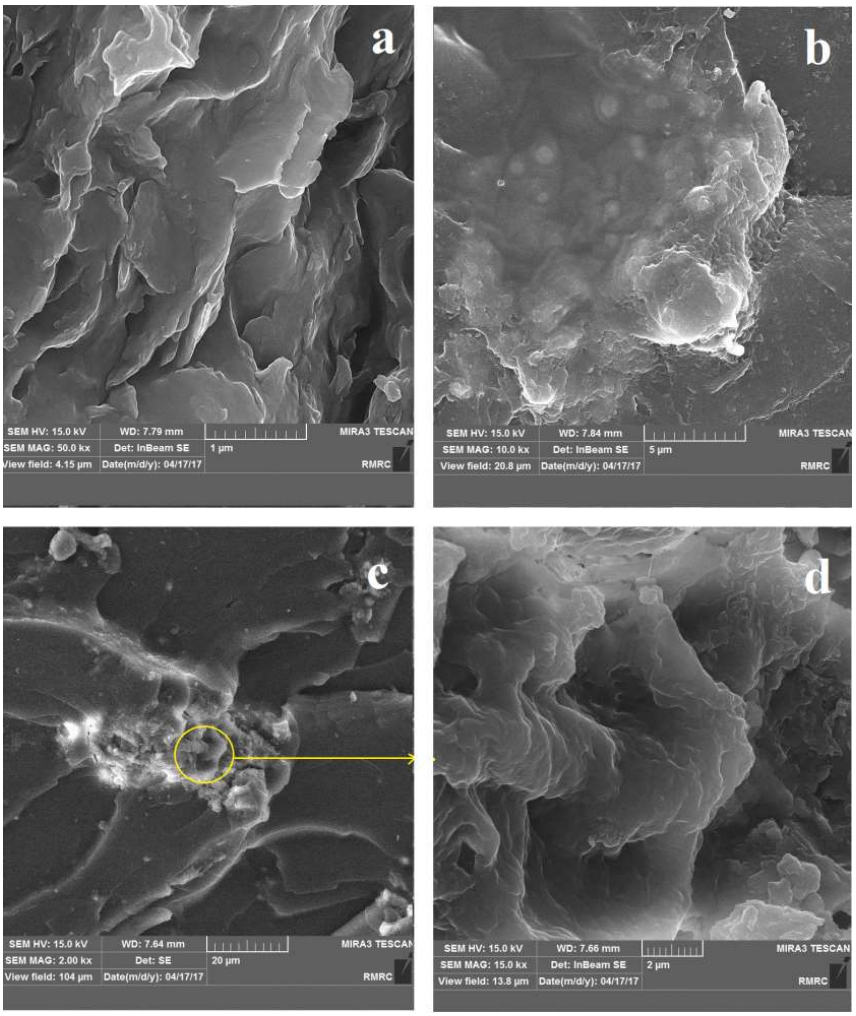

Fig. 14. FESEM micrographs of the fracture surfaces of FMLs containing (a) the 3 wt.% modified nanoclay, (b) the 5 wt.% modified nanoclay, and (c, d) the 3 wt.% unmodified nanoclay.

4. Conclusion

The effect of nanoclay addition by 1, 3 and 5 wt.% and the influence of the 3 wt.% unmodified nanoclay on the flexural and impact behavior of basalt fibers/ epoxy-aluminum

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2024-T3 laminated composites were investigated. The results could be summarized as follows:

a) The maximum effect of surface- modified nanoclay on the flexural strength could be achieved at 3 wt.%. The flexural strength was elevated by 52% and 31%, as compared to the nanoclay-free and unmodified nanoclay samples, respectively; yet, an uprising trend for the flexural modulus was observed with the increase in the amount of the modified nanoclay.

b) Due to the improvement of the epoxy /basalt fibers interface by adding the modified nanoclay up to 3 wt.%, an increase could be obtained in the flexural strength. On the other hand, the rise in the flexural modulus caused a reduction of shearing stress in the composite/aluminum sheet interface that led to enhancing the flexural strength as well.

c) A weaker flexural and impact property in the 3 wt.% unmodified nanoclay was achieved in comparison to the surface- modified ones; according to the microscopic images, this mostly resulted from the agglomeration of the clay laminates. Furthermore, the study of the fracture surface in the samples revealed a significant enhancement in the epoxy/basalt fibers interface properties in those samples containing nanoclay.

d) The most efficient percentage of surface modified nanoclay addition on the energy absorption related to the 3 wt.% addition. The energy absorption led to 10% and 12% improvement, as compared to nanoclay-free and unmodified nanoclay samples, respectively. Similar findings also confirmed that the destruction area caused by the impact in the 3 wt.% surface- modified nanoclay sample had the largest destruction area among all samples.

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