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Investigating the High Velocity Impact Behavior of the Laminated Composites of Aluminum/Jute Fibers- Epoxy Containing Nanoclay Particles

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Abstract

In this research work, the effect of adding nanoclay on the high velocity impact behavior of fibers metal laminates (FMLs) of aluminum- Jute fibers/epoxy was investigated. To do so, the nanoclay particles with different percentages (0, 1, 3 and 5 wt.%) were firstly added into the epoxy matrix. In the following, the FMLs with the configuration of 0/90/0/90/0 were made by hand lay-up method. After that, the fabricated samples were tested by high velocity impact test. In order to investigate the effect of nanoparticles on the impact properties, the field emission scanning electron microscope (FESEM) was used. The obtained results showed that the samples with 0, 1, 3 and 5 wt.% had the limit velocity of 81.9, 83.7, 87.4 and 85.4 m/s, respectively. The limit velocity increment in these samples by adding nanoclay were 2.2, 6.7 and 4.3 %, respectively. Also, the obtained results showed that by adding the 1, 3 and 5 wt.% nanoclay, the absorbed energy was improved about 4.5, 14 and 8.8 %, respectively. The sample with 3 wt.% nanoclay had minimum delamination length between aluminum shell and composite core, which was due to the effect of nanoclay in the interface adhesion of core-shell. The microscopically analysis showed that the nanoclay increased the fibrillation of Jute fibers as one of the absorbed energy mechanisms in the Jute fibers. Also, the agglomeration

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phenomenon in the sample with the 5 wt.% nanoclay was illustrated, which was the decreasing factor of its impact properties, as comparison with sample with 3 wt.% nanoclay.

Keywords: Fibers metal laminate, Nanoclay, Jute fibers, High velocity impact

1. Introduction

Nowadays, using the composite materials in the different industries is very common, due to their specific features like high strength and stiffness to weight ratio [1,2], high resistance to fatigue and corrosion [3,4], so that they have been replaced with some metallic parts. However, they have disadvantages such as low working temperature, high thermal expansion coefficient and water absorption from environment. The simultaneous usage of composite and metals was the interesting idea for emitting these disadvantages. This innovative idea was caused to fabricate the new kind of composite materials as the name of fiber metal laminates (FMLs) [5].

Adding the nanoparticles such as carbon nanotubes (CNTs) [6], graphene nanoplatelets (GNPs) [7], graphene oxide [8], nanosilica [9] and nanoclay [10] into the polymeric composites is one of the interest subjects that a wide investigation has been carried out about that. One of the important goals for adding those is improving the mechanical properties of composites. Therefore, in the recent years, using these nanoparticles into the FMLs is one of the newest researches, which has attracted many attentions of researchers about that. Khurram et al. [11] investigated the effect of CNTs on the adhesion between aluminum and glass fibers epoxy composite. The obtained results showed that the CNTs were caused to improve the adhesion between those by creating the nano-mechanical locks. The optimum percentage of CNTs was reported 2.5 wt.% by these researchers.

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In the study of Bahari et al. [12], the effect of adding modified nanoclay particles on the impact and flexural properties of FMLs were investigated. In this research, the 2024 aluminum as skin and basalt fibers- epoxy composite containing nanoclay as core of FMLs were used. They reported that adding the 3 wt.% modified nanoclay was improved the fracture energy and flexural strength of FMLs. Pol et al. [13] surveyed the high velocity impact behavior of FMLs containing glass fibers and nanoclay particles. The obtained results showed that the FMLs containing the 3 and 10 wt.% nanoclay could absorb the energy of impactor with the rate of 134 and 169 m/s, respectively.

Haro et al. [14] investigated the effect of adding micro and nano fillers of aluminum, silicon carbide and colloidal silica on the energy absorption of reinforced FMLs. The selected shell and core in the FMLs were aluminum 5086 and Kevlar fibers- epoxy composite, respectively. The reported results showed that the nano fillers by adhering the fibers together increased the absorbed energy of FMLs, but the micro fillers by filling the cavities were caused to strongly enhance the absorbed energy.

The natural fibers have a good potential for replacing with synthesizing fibers like glass or Kevlar fibers because the composites have increasingly growth and using the synthesizing fibers into them has created environmentally concerns. Therefore, replacing the natural fibers with the synthesizing fibers is so necessary [15-17]. Similar the other composite structures, using the natural fibers into FMLs is inevitable. Therefore, the efforts, though limited in scale, have been done for using the natural fibers into FMLs. Malingam et al. [18] firstly fabricated the hybrid glass-kenaf reinforced aluminum laminate with the different orientation of kenaf fibers and then investigated the tensile and impact behaviors of FMLs. In the similar research, Feng et al. [19] surveyed the fatigue behavior of those FMLs.

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The Jute fibers can be good candidate for replacing with the synthesizing fibers, due to low production costs and very accessibility [20]. Therefore, these natural fibers can be used into the composite structures especially in the FMLs. The eco-friendly FMLs is the name of this kind of FML structures [21]. These structures can be used in the automotive industries, due to low cost, low density and biodegradability of their fibers. In this regard, the present work aims to fabricate the FMLs structures with aluminum as shell and Jute fibers- epoxy composites containing various percentages of nanoclay as core. After that, the impact behaviors of these FML structures are investigated and compared.

2. Experimental

2.1. Raw Materials

The epoxy resin (KER 828) and triethylenetetramine (TETA) polyamine hardener were used as matrix. The plain weave Jute fibers with the surface density of 350 g/m^2, supplied by Taizhou (China), were used. 2024-T3 aluminum sheets with thickness of 0.5 mm were used as metal layers in the FMLs structures. The Cloisite 5A nanoclay with the 2030 nm interlayer distance, supplied by BYK (German), was selected in the current work. The chemical name of this nanoclay is Bis(hydrogenated tallow alkyl)dimethylammonium bentonite. For surface modification of aluminum layers and Jute fibers, the commercial grade of sulfuric acid (98 wt.%, H_2SO_4), potassium dichromate ($K_2Cr_2O_7$) and sodium hydroxide (NaOH) were used.

2.2. Surface Modification

It is worth noting that the Jute fibers have hydrophilic natures, so that, the adhesion between those and epoxy resin is very weak. Therefore, the surface modification for reducing the hydrophilic nature of those is necessary. Based on previous work [22], the Jute fibers were

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soaked into the 1 M NaOH solution for 2 h and after that they were washed with the deionized water.

To improve the adhesion between aluminum layers and composite, the surface modification of aluminum was done as per previous work through forest products laboratory (FPL) etch method [23]. To do so, firstly the aluminum layers were soaked into the 1 M NaOH for 5 min. After that, they immersed into the FPL solution for 45 min in the temperature of 60 ^{o}C. The ingredients of FPL solution were 350 g sulfuric acid, 35 g potassium dichromate and 2 L deionized water.

2.3. FMLs Fabrications

To fabricated the FMLs containing the various weight percentages of nanoparticles (0, 1, 3 and 5 wt.%), the nanoclay were firstly mixed with the epoxy by three-blade mechanical stirrer (Finetech, Korea) for 5 min. After that, for removing the agglomerated nanoclay particles, the mixture was sonicated by ultrasonic device (Fapan, Iran) with the power of 120 W for 15 min. After 5 min, the mixture was sonicated again. Then, the mixture was stirred by mechanical stirrer for 30 min to achieve the homogeneous mixture. In the final step, the sonication was performed for 2 times again. It is worth noting that during the sonication process, the mixture was put into the ice bath for reducing the temperature. In this research, the core of FMLs was the Jute- epoxy composite with the configuration of 0/90/0/90/0. To fabricate the FMLs with the dimensions of 13×13 cm^2, firstly the aluminum layer was wet with the mixture of epoxy-nanoclay. After that, the composite core was fabricated by hand lay-up method [24]. Then the upper layer of FMLs (aluminum sheet) was set on the composite core. In the following, the FMLs were put into the mold and pressed by hydraulic press in the pressure of 4 MPa for one

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day to cure the epoxy at the temperature of 25 ºC. To post cure the FLMs, they set in the ambient temperature for 7 days, as recommendation of producer.

2.4. High Velocity Impact Test

The high velocity impact test was performed by a gas gun machine with the initial velocity of 97 m/s. The schematic of this device has been illustrated in Figure 1. The used impactor was hemispherical shape which the length and weight of that were 4 cm and 27 g, respectively. The limited velocity and absorbed energy can be calculated by equations of (1) and (2) [25]:

$$V_{50} = \sqrt{V_0^2 - V_r^2} \tag{1}$$

$$E_a = \frac{1}{2} m_i (V_0^2 - V_r^2) \tag{2}$$

where V_{50}, V_0, V_r, m_i and E_a were limited velocity, initial velocity, residual velocity, impactor mass and absorbed energy, respectively.

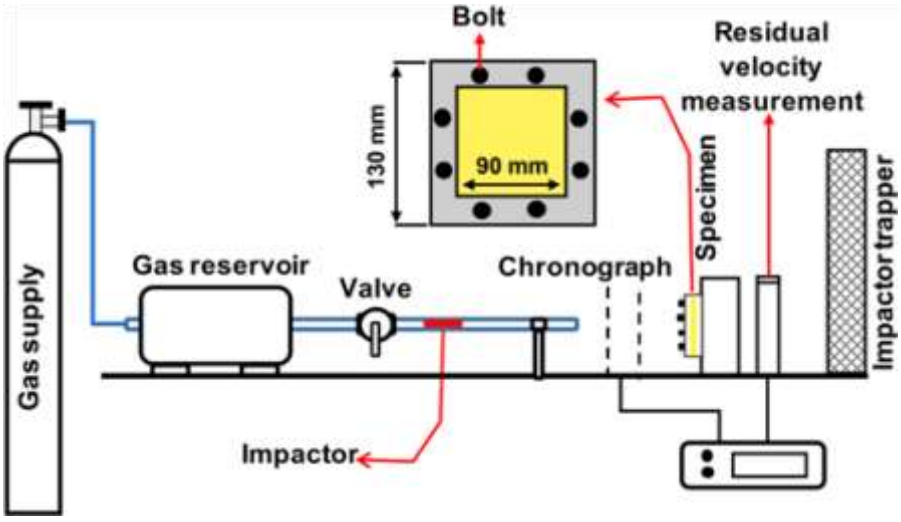

Figure 1. Schematic diagram of high velocity impact device.

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2.5. Microstructural Characterization

In the current study, the optical microscopy (OM), Meiji Techno, Japon, was used for image analyzing of surface modified aluminum layers. Also, for investigating the fracture surface of composite core and aluminum layers and interaction between nanoclay and epoxy matrix, the field emission scanning electron microscopy (FESEM), Mira3 Tescan-Czech, was used.

3. Results and Discussion

3.1. Surface Modification of Aluminum

Figure 2 shows the un-modified and modified surface of aluminum sheet. It can be seen that the surface of modified aluminum has changed and the areas containing the cavities have been created on that. The existence of these cavities is caused to create the mechanical locking in the FML structures [26]. Figure 3 represents the FESEM analysis of aluminum sheet in the FMLs containing 3 wt.% nanoclay after impact test. As specified by the arrows, a part of epoxy has diffused into the created cavities in the aluminum surface.

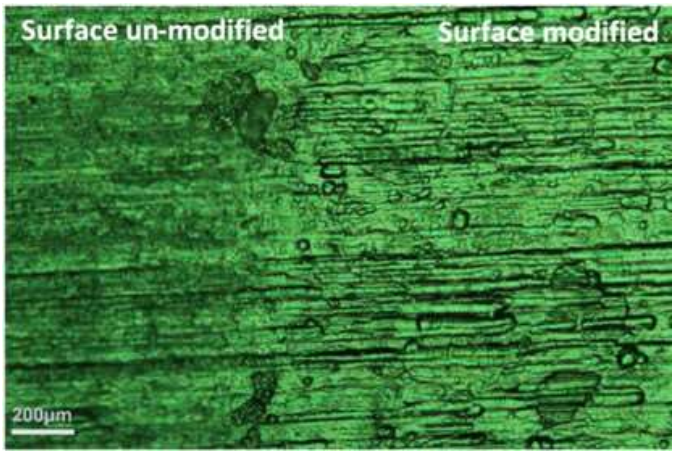

Figure 2. Unmodified and modified surface of aluminum after FPL etch by OM.

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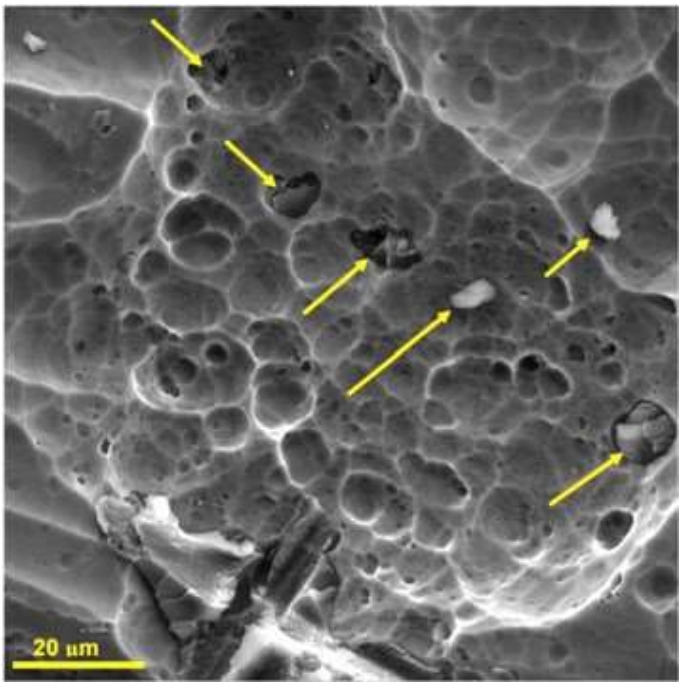

Figure 3. Structure of aluminum surface after surface modification and diffusing the epoxy into that.

3.2. High Velocity Impact Test

The obtained results from the impact test has been listed in Table 1. Based on the Table 1 and percentage of used nanoclay, the Figures 4 and 5 were drawn. Figure 4 shows the residual velocity of impactor in the different FML samples. Based on this figure, the sample with 0 wt.% nanoclay had 52 m/s residual velocity, which was the maximum of that. By adding the 1 wt.% nanoclay, this velocity was reduced to 49 m/s. This means that the residual velocity had 5 % reduction. By reaching the percentage of nanoclay to 3 wt.%, this velocity with 19% reduction was reached to 42 m/s. This reduction was the maximum reduction, in comparison with other samples. In the FMLs containing 5 wt.% nanoclay, the residual velocity was about 46 m/s. By comparing with the without nanoclay sample, the 11.5 % reduction in the residual

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velocity was observed, whereas this sample had the 9.5 % increment in residual velocity, as comparison with FMLs containing 3 wt.% nanoclay.

Table 1. Obtained result from high velocity impact test of FMLs

Nanoclay content (wt.%)	Residual velocity (m/s)	Limit velocity (m/s)	Absorbed energy (J)	Specific absorbed energy (J/kg)
0	52	81.9	90.5	982
1	49	83.7	94.6	1045
3	42	87.4	103.2	1093
5	46	85.4	98.5	1062

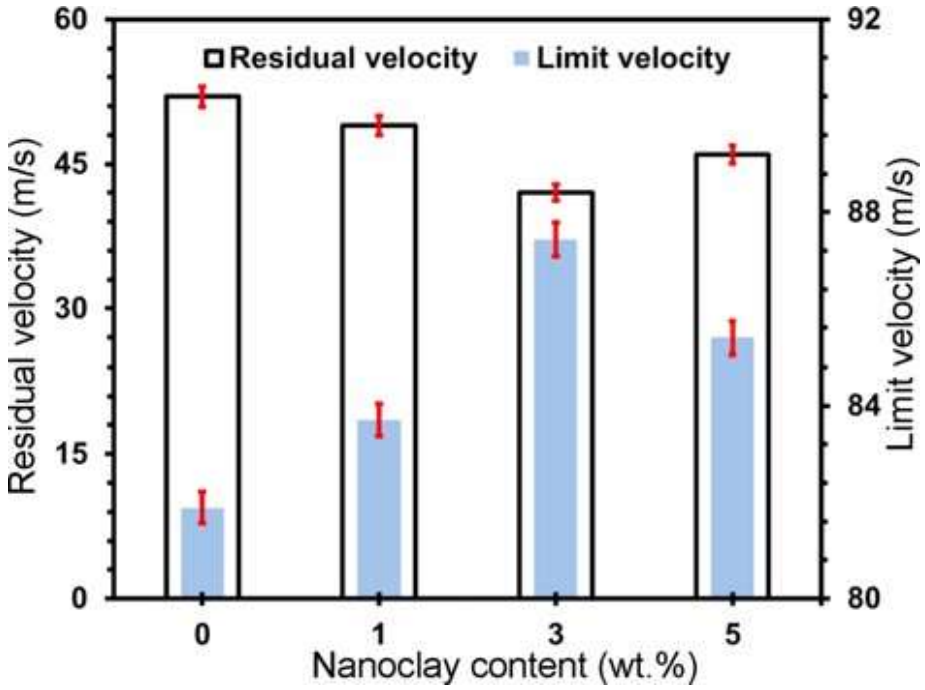

Figure 4. Effect of the nanoclay content on the residual and limit velocity of FMLs.

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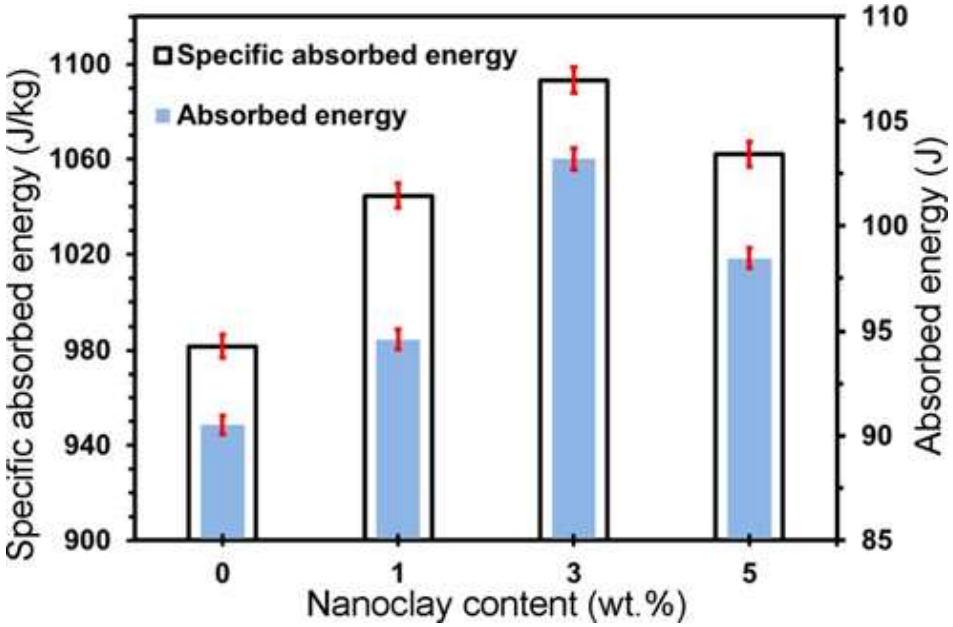

Figure 5. Effect of the nanoclay content on the absorbed energy and specific absorbed energy of FMLs.

Based on Figure 4, it can be seen that the without nanoclay sample with 81.9 m/s limit velocity had the minimum limited velocity, as comparison with other samples. Samples containing 1, 3 and 5 wt.% nanoclay had 83.7, 87.4 and 85.4 m/s limit velocity, respectively. The increment in these samples by adding nanoclay were 2.2, 6.7 and 4.3 %, respectively. According to the Figure 4, it can be observed that the sample containing 3 wt.% nanoclay had the maximum limit velocity. Based on the other literatures [27], the increment of limit velocity in composites is related to control crack growing and propagating by nanoclay particles. It is worth noting that the nanoclay into the composites can be inform of exfoliated or intercalated. By applying the shear force through the three-blade mechanical stirrer, the silicate layers of nanoclay are separated and the exfoliated structure is formed. When the micro-cracks reach the nanoclay, the crack propagation mode is changing from planar to tortuous. This phenomenon wastes the propagation energy of microcracks. So that, the microcracks need higher level of

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energy for propagation. In the exfoliated structure, the higher silicate layers change the crack propagation than intercalated structure. Therefore, the exfoliated form of nanoclay has higher improvement in mechanical properties than the intercalated form [28,29].

Figure 5 shows the absorbed energy and of specific absorbed energy (SAE) in different samples. To more accurate of results, the SAE was calculated, which is the absorbed energy per the weight of sample. In this figure, the sample with 3 and 0 wt.% nanoclay had the maximum and minimum absorbed energy, as compared with other samples. The obtained results showed that by adding the 1, 3 and 5 wt.% nanoclay, the absorbed energy was improved about 4.5, 14 and 8.8 %, respectively.

3.3 Microscopic Analysis

Figure 6 represents the FESEM analysis of composite core of FMLs containing different percentages of nanoclay. Given up Figure 6a, it can be understood that the adhesion of epoxy to the Jute fibers is weak, so that, the remained resin cannot be seen on the surface of the Jute fibers. This can be attributed to the hydrophilic nature of the Jute fibers [30]. The adhesion between fibers and matrix has an important role on the mechanical properties and transferring the applied loads, so that, having the weak adhesion on the interface of those reduces the mechanical properties. By adding the 1 wt.% nanoclay into the matrix, the adhesion between fibers and epoxy was improved, which can be seen in Figure 6b. By increasing the percentage of nanoclay up to 3 wt.%, the adhesion between of those is strongly improved, so that, the Jute fibers stick together, which can be observed in Figure 6c.

Adding the 5 wt.% nanoclay was caused to create the agglomerated nanoclay, which has been specified by the arrow in Figure 7. Generally, the cracks can cross in the weakest area in the composites. In the agglomerated area, the resin cannot wet all nanoparticles. Therefore,

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they have weak interface and cracks can easily cross between them, which reduces the mechanical properties of composites. Figure 6d shows the fracture surface of FMLs containing 5 wt.% nanoclay after impact test. Considering with Figure 6d, it can be seen that the adhesion between Jute fibers and epoxy has been reduced. So that, the fibers pull out and imprint phenomena on the matrix are seen. The imprint phenomenon is created after the pulling out the Jute fibers in the interface of Jute-epoxy. It is worth noting that the obtained results has good conformity with other researches in the field of composites containing nanoclay particles [12].

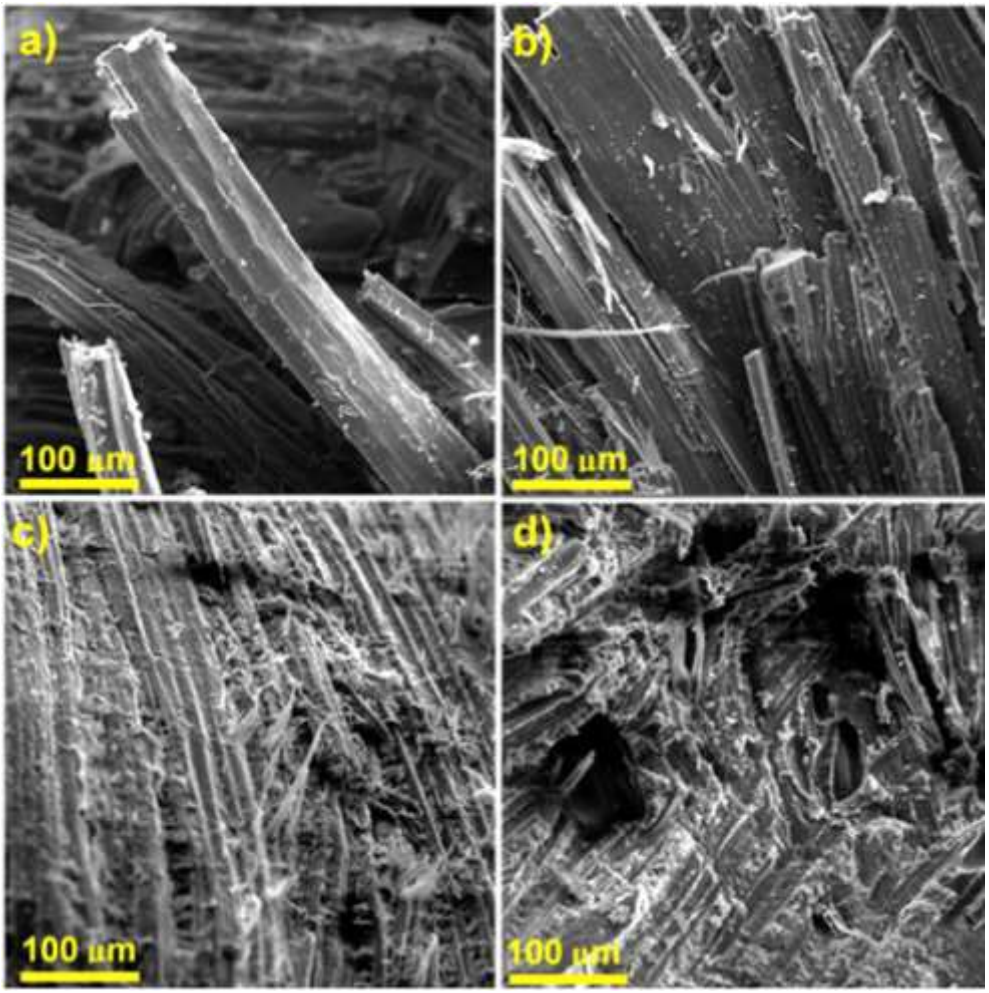

Figure 6. FESEM analysis from the fracture surface of FMLs after high velocity impact test containing nanoclay; (a) 0, (b) 1, (c) 3, and (d) 5 wt.%.

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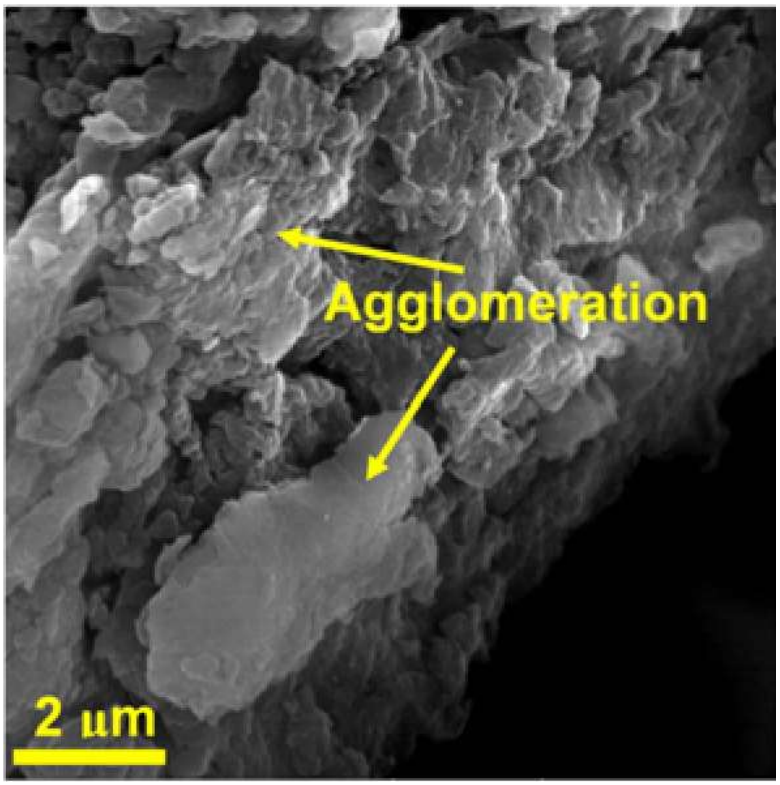

Figure 7. Formed agglomerations in the samples containing 5 wt.% nanoclay.

One of the mechanisms for absorbing the energy in composites is fibrillation of fibers especially natural fibers [31]. By creating the fibril from the fibers, the energy of impact can be wasted. Figure 8 illustrates the effect of nanoclay on the fibrillation of Jute fibers. Based on the Figure 8a, it can be seen that the fibrillation of the Jute fibers is very low. Whereas, by adding the 1 wt.% nanoclay, this phenomenon has been increased. The maximum fibrillation belongs to the FMLs containing 3 wt.% nanoclay. Also, in Figure 8d, it can be observed that the fibrillation of the Jute fibers has been reduced, as compared with sample containing 3 wt.% nanoclay. The fibrillation behavior into the FMLs had good agreement with the impact behavior of those. This behavior can be attributed to the effect of nanoclay on the crack propagation into the matrix. The microcracks tend to cross the weakest area into the composite.

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By adding the nanoclay, the matrix was reinforced. Also, the adhesion between Jute fibers and epoxy was improved. Therefore, the microcracks crossed in the weakest area into the Jute fibers, so that, it was caused to the separate some part of fibers (fibrillation phenomenon).

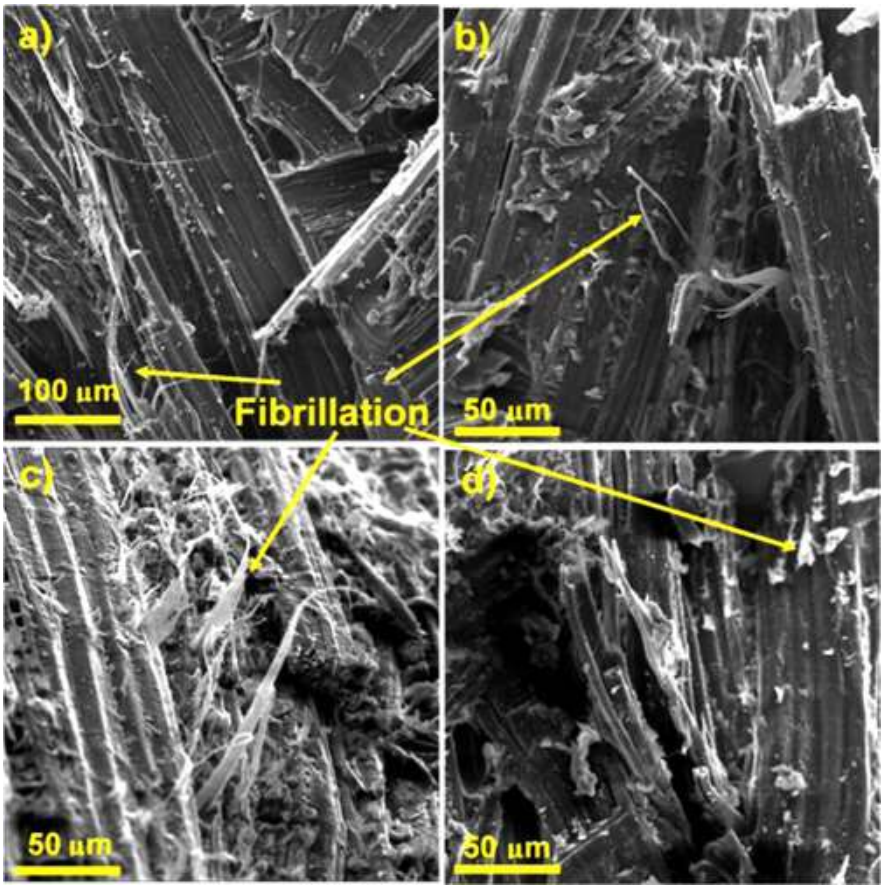

Figure 8. FESEM analysis from fibrillation phenomenon of Jute fibers; (a) 0, (b) 1, (c) 3, and (d) 5 wt.%.

One of the important factors in the FMLs is the adhesion between metal and composite layers. Therefore, investigating the effect of nanoclay on the interface of aluminum and composite is so necessary. Figure 9 depicts the aluminum surface in FMLs, after the impact test. According to Figure 9a, it can be seen that in the sample without nanoclay, only a little of epoxy has been adhered on the surface of aluminum. By adding various percentages of

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nanoclay into the epoxy, in addition to adhering the epoxy, the fibers imprint and fibrillation phenomena can be seen. This means that adding the nanoclay improved the adhesion between the aluminum and composite layers, so that, it can be reduced the delamination length between of those. The reason of that is changing the failure mechanism from the adhesive (crossing the crack among interface of aluminum- epoxy) to cohesive failure (crossing the crack among epoxy matrix). This changing the failure mechanism was caused to improve the load barring from aluminum to the composite layer, consequently, improved the impact properties of FMLs. The results of limit velocity and absorbed energy confirmed this assumption. The specified arrows in Figure 10 clearly explain the effect of nanoclay on the surface of aluminum layer. Based on this figure, it can be seen that the epoxynanoclay mixture diffuses into the created cavities by the FPL etching. In other words, in the FMLs without nanoclay, the epoxy only diffuses into the aluminum layer and creates mechanical locking. Adding the nanoclay into the epoxy was caused to form the nanocomposite mechanical locks, which had higher mechanical properties, as compared with epoxy mechanical locks. Therefore, these nanocomposite mechanical locks were caused to improve the adhesion between aluminum shell and composite core.

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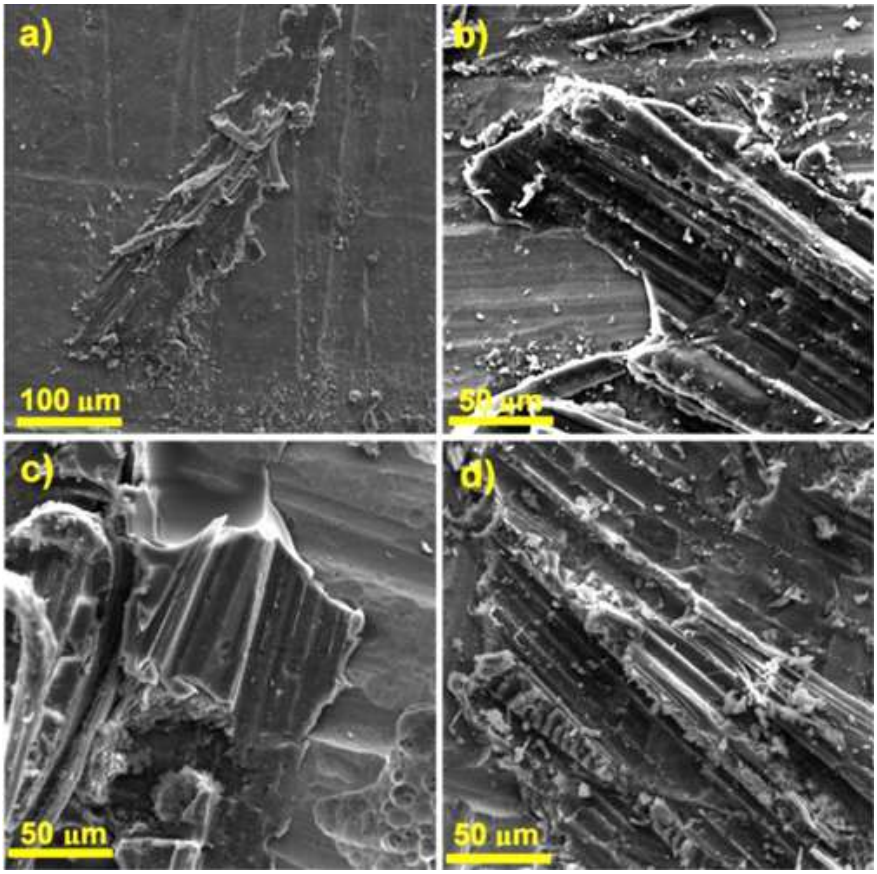

Figure 9. FESEM analysis from adhesion between aluminum shell-composite core in FMLs; (a) 0, (b) 1, (c) 3, and (d) 5 wt.%.

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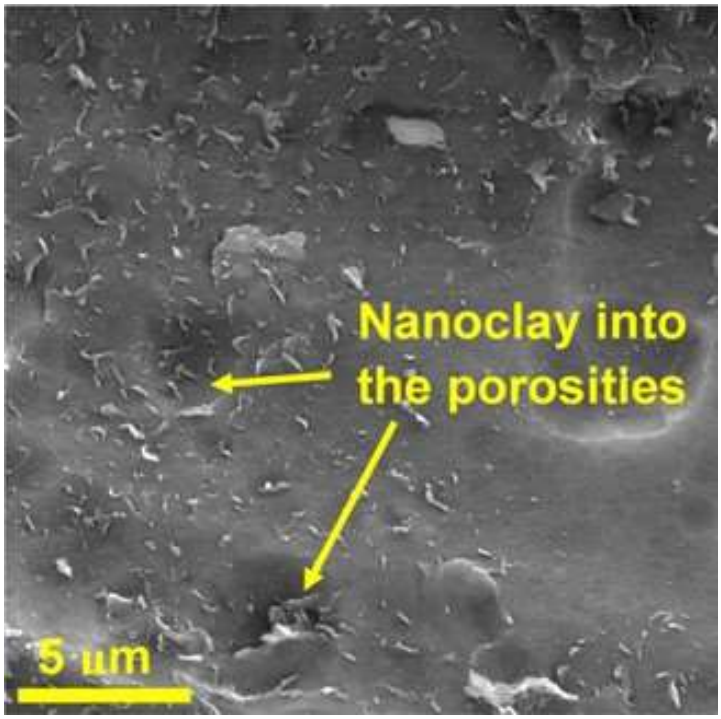

Figure 10. Diffusing nanoclay into the porosity structure of aluminum surface.

3.4. Macroscopic Analysis

Figure 11 shows the damaged area in the FMLs containing various percentages of nanoclay. By measuring the damage area, the samples containing 0, 1, 3 and 5 wt.% nanoclay had 5.3, 6.6, 7.6 and 7.3 cm^2 damaged area, respectively. Based on these results, the FMLs containing 3 wt.% had the maximum damaged area. This means that by increasing the damaged area, the FML can absorb the higher energy of impactor, consequently, the residual velocity of impactor is reduced. This trend can be attributed to the effect of intercalation and exfoliation structures of nanoclay in the epoxy matrix, which previously mentioned by other literatures [32].

Figure 12 depicts the delamination length of aluminum layer from composite core, after impact test. Based on this figure, the delamination length of without nanoclay sample was 5

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cm. By adding the 1 wt.% nanoclay into the FMLs, the delamination length was reached to 6 cm. The similar behavior was reported by other researchers in the FMLs structures [33]. The surveys show that the crack propagates from the weakest area. Also, the adding the nanoclay is caused to improve the adhesion of the Jute fibers and epoxy. For this reason, the crack has been grown from the interface of aluminum and composite core, consequently, the delamination length has been increased. The obtained results for sample containing 3 wt.% nanoclay showed that the delamination length had a reducing trend and reached to 4.5 cm. It can be attributed to the interaction between nanoclay and surface modified aluminum. As previously mentioned, the FPL treatment created the porous structures on the surface of aluminum. This porous structures were filled by epoxy resin during the fabrication process, so that, the mechanical locks were created in the interface of aluminum and composite. Adding the nanoclay was caused to fabricate the nanocomposite mechanical locks. These nanocomposite locks were influenced on the crack behavior, so that, they reduced the delamination length in FMLs containing 3 wt.% nanoclay. The obtained delamination length for sample containing 5 wt.% was about 8 cm. This increment can be attributed to the agglomerations of nanoclay particles, which acted as weakest area for propagating the cracks.

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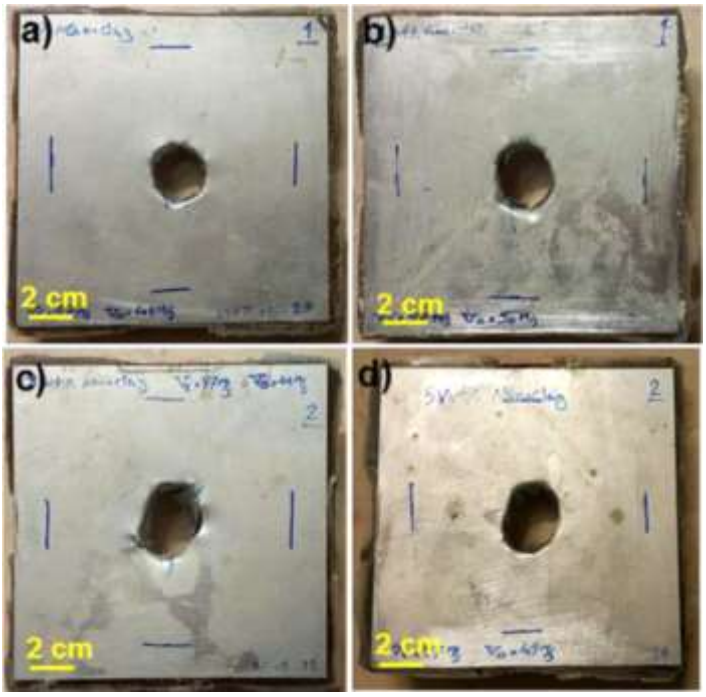

Figure 11. Damaged area in the FMLs containing nanoclay; (a) 0, (b) 1, (c) 3, and (d) 5 wt.%.

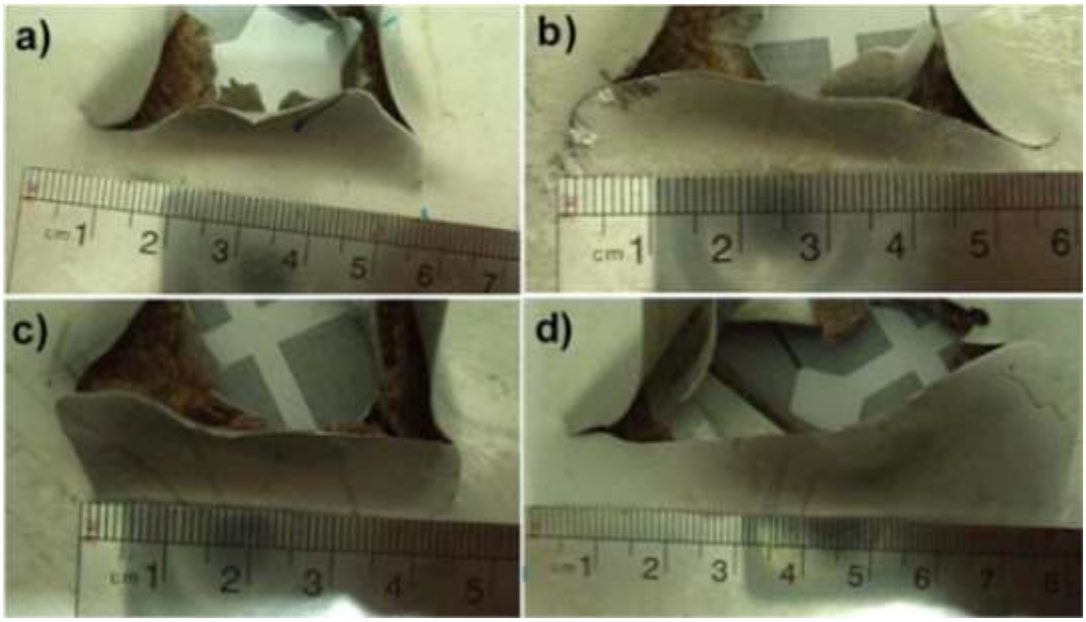

Figure 12. Delamination length in the FMLs containing nanoclay; (a) 0, (b) 1, (c) 3, and (d) 5 wt.%.

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4. Conclusion

In the present study, the effect of various percentages of nanoclay (0, 1, 3 and 5 wt.%) on the impact behavior of fibers metal laminates (FMLs) of aluminum- Jute fibers/ epoxy was investigated. The results of this study can be summarized here:

1. The FMLs containing the 3 wt.% nanoclay with the 87 m/s limit velocity, 103.2 J absorbed energy, 1093 J/kg specific absorbed energy and 7.6 cm^2 damaged area had the maximum impact properties, as compared with the other FMLs.
2. The FMLs containing 3 wt.% nanoclay had 4.5 cm delamination length, which was minimum delamination length, due to the effect of nanoclay on the improvement of adhesion between aluminum layer and composite core.
3. The microscopically investigations showed that improving the adhesion between the Jute fiber and epoxy was caused to increase the fibrillation of the Jute fibers as absorbed energy mechanism in the FMLs.
4. Diffusing the nanoclay particles into the created cavities on the surface of aluminum was caused to create the nanoclay- epoxy nanocomposite mechanical locks, which improved the adhesion between aluminum and composite.

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