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Interlaminar shear strength and tensile properties of environmentallyfriendly fiber metal laminates reinforced by hybrid basalt and jute fibers

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Abstract

The mechanical properties of environmentally-friendly fiber metal laminates (EFFMLs) with jute (J)- basalt (B) fibers as a hybrid reinforcement and aluminum (Al) 2024-T6 as a skin, as well as an epoxy serving as a matrix, were investigated by employing tensile and interlaminar shear tests. Four hybrid structures including Al/J/J/B/ B/J/J/Al and Al/B/B/J/J/B/B/Al as the sandwiched structures and Al/J/B/J/B/J/B/Al and Al/B/J/B/J/B/J/Al as the intercalated structures were fabricated. The results showed that the jute fibers sandwiched by the basalt fibers had the highest tensile strength, elastic modulus and interlaminar shear strength (ILSS) values; these were 213 MPa, 39.5 GPa, and 16.67 MPa, respectively. In contrast, these values were 139MPa, 29.6 GPa and 10.3 MPa for the tensile strength, elastic modulus, and ILSS of the basalt fibers sandwiched by the jute fibers, respectively. Microstructural investigations also revealed that the jute fibers had some weak bonding with the aluminum layers, whereas the basalt fibers had the strong one with them. It was also found that the lack of diffusion caused the presence of empty spaces between the jute fibrils, and these defects were the main cause of the decrease in the mechanical properties.

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Keywords: Hybrid composites, Mechanical properties, Fiber metal laminate, Basalt fibers, Jute fibers

1. Introduction

Fiber metal laminates (FMLs) are new high-performance materials used in aerospace, marine and civil industries due to such superior properties as lightweight, high strength to weight ratio, excellent fatigue performance and impact resistance [1,2]. FMLs are hybrid materials fabricated by the thin layers of metals and fiber-reinforced composites [3,4]. The thin metal layers of these materials can be aluminum, magnesium, titanium or some grades of steels [5,6]. It is worth noting that the composite layers of FMLs can be reinforced by glass, carbon or aramid fibers in different thermoset or thermoplastic polymers. The most famous FMLs used in the industries are GLARE, CARAL and ARALL, which are fabricated by the glass, carbon and aramid fibers placed between the aluminum sheets, respectively [7]. The reduction of the product costs and the enhancement of the mechanical properties are the most attractive issues in the use of other fibers in the FMLs structures.

In the past few years, the use of the eco-friendly fibers as the reinforcement in the composites has received much attention [8]. One of the best candidates for using these fibers instead of the aramid, carbon or glass fibers is the basalt fibers. The basalt fibers are inorganic fibers with chemical compositions to similar to those of the glass fibers [9], but with higher mechanical properties, and chemical or thermal stability [8]. Up to now, the use of basalt fibers as the reinforcement in the FMLs structures has been considered by a limited number of researchers. Khalili et al. [10], for instance, examined the mechanical behavior of composites reinforced with the basalt fibers in the FMLs structures. They also studied the charpy impact behavior of these structures in another work [11]. Ferrante et al. [12] also investigated the impact properties of these FMLs. Further Kuan et al. [13] compared the fracture behavior of

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different FMLs reinforced by the hemp, flax and basalt fibers, introducing them as environmental-friendly fiber metal laminates (EFFMLs).

Many attempts have been made to use other natural fibers such as banana, flax, sisal and jute fibers in the composites [14,15]. Nevertheless, unlike the basalt fibers, these fibers have not been promising, mainly due to the poor mechanical properties, low thermal and chemical stability, and hydrophilic issues. The jute fiber is one of the natural fibers with a high hydroxyl group in its cellulose structure, and poor compatibility with the polymeric matrices [16]. It is well known that an interface between the fiber and the matrix can have a significant effect on the mechanical properties of the composites. Therefore, to improve the interface bonding between the jute fibers and the polymeric materials, the physical or chemical surface modification of the fiber surfaces is necessary [17].

Sinha et al. [18] used some alkaline treatment to modify the jute surface by the 5 wt% NaOH solution. They reported that the hemicellulose and lignin were removed after alkaline treatment and a rough surface morphology could be obtained. They verified their results by SEM observations. Sever [19] also used some oligomeric siloxane for surface modification. The obtained results showed that the interlaminar shear strength of the composites was improved by approximately 98%. The chemical modification with fatty acids is another technique employed by Corrales et al. [20]. Seki et al. [16] also investigated the effect of plasma treatment on the surface of the jute fibers. They reported the improvement in the flexural and tensile strength of the composites by enhancing the fiber/matrix adhesion.

In light of the above, the aim of this work was to investigate the mechanical properties of the EFFMLs structures reinforced by the jute (natural fibers) and basalt (mineral fibers) hybrid fibers in the epoxy matrix. These fibers were stacked with different sandwiched and

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intercalated stacking sequences according to the previous works done on the hybrid composites [21–23].

2. Materials and experimental procedures

2.1. Materials

Al 2024-T6 sheets with the thickness of 0.3 mm were used as the skin of EFFMLs. In order to fabricate the composite core, the basalt and jute fibers as the reinforcement and Epone 828 epoxy resin and triethylenetetramine hardener as the matrix were used. The basalt fabric, supplied by Basaltex (Belgium), had an atlas weave with a silane agent (the surface density 350 g/cm^2). Also, the jute fabric, supplied by Taizhou (China), had a plain weave (the surface density of 350 g/cm^2). In order to modify the surfaces of aluminum sheets and jute fibers, the commercial grades of sodium hydroxide (NaOH), sulphuric acid (98 wt %, H_2SO_4) and potassium dichromate ($K_2Cr_2O_7$) were used.

2.2. Surface modification

To increase the interfacial bonding between the aluminum sheets and the epoxy matrix, the aluminum sheets needed the surface modification. One of the simplest methods for the surface modification is forest products laboratory (FPL) etching. The FPL solution was prepared by the Eickner method [24]. In the first glass container, 35 g sulphuric acid 98 wt% was diluted into 100 g deionized water. In the other container, 3.5 g potassium dichromate was dissolved in 100 g deionized water. After increasing the temperature to 60 °C, the diluted sulphuric acid was added to the potassium dichromate solution.

In the first step, the aluminum sheets were cleaned by acetone to remove greases and surface contaminations. Then, the aluminum sheets were immersed in the 4 wt% NaOH

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solution for 10 min to remove the oils and greases which had not been removed by acetone [25]. Afterwards, the aluminum sheets were washed by the deionized water. In the next step, the aluminium sheets were immersed in the FPL solution. The optimized etching time of the aluminium sheets by the FPL solution was 30–45 min. In the final step, the aluminium sheets were washed by the deionized water and then dried at the ambient temperature.

To decrease the hydrophilicity of the jute fibers, the alkaline treatment was accomplished. Accordingly, the jute fibers were immersed in the 5 wt% NaOH solution for 2 h at the ambient temperature. After that, these fibers were washed several times with the deionized water and then dried in a vacuum oven at the temperature of 65 °C for 24 h. Scanning electron microscopy (SEM- Vega/Tescan LMU, Czech) was used to verify the jute fibers and aluminum surface modification, and to observe the distribution of the fibers in the sample cross sections.

2.3. Composite preparation

Based on the literature, the modified structures in the hybrid composites are sandwiched and intercalated ones [23,26]. Therefore, as can be seen in Fig. 1, two sandwiched and two intercalated configurations with different stacking sequences of the jute and basalt fibers were selected as the core in the FML structures. In the first step, the FMLs were fabricated through a hand lay-up method with the dimensions of 30 cm × 20 cm and pressed. Then, the samples were allowed to cure in room temperature for 24 h under the pressure of 4 MPa. In order to do post curing, the FMLs were placed in the oven for 2 h at the temperature of 100 °C according to producer recommendation. The measured weight fraction of the jute and basalt fibers to the epoxy resin was 50:50 wt %. Eventually, for each test, the samples were cut by water-jet. It should be noted that the thickness of each layer of aluminum, jut and basalt fibers were 0.5, 0.32 and 0.8 mm, respectively.

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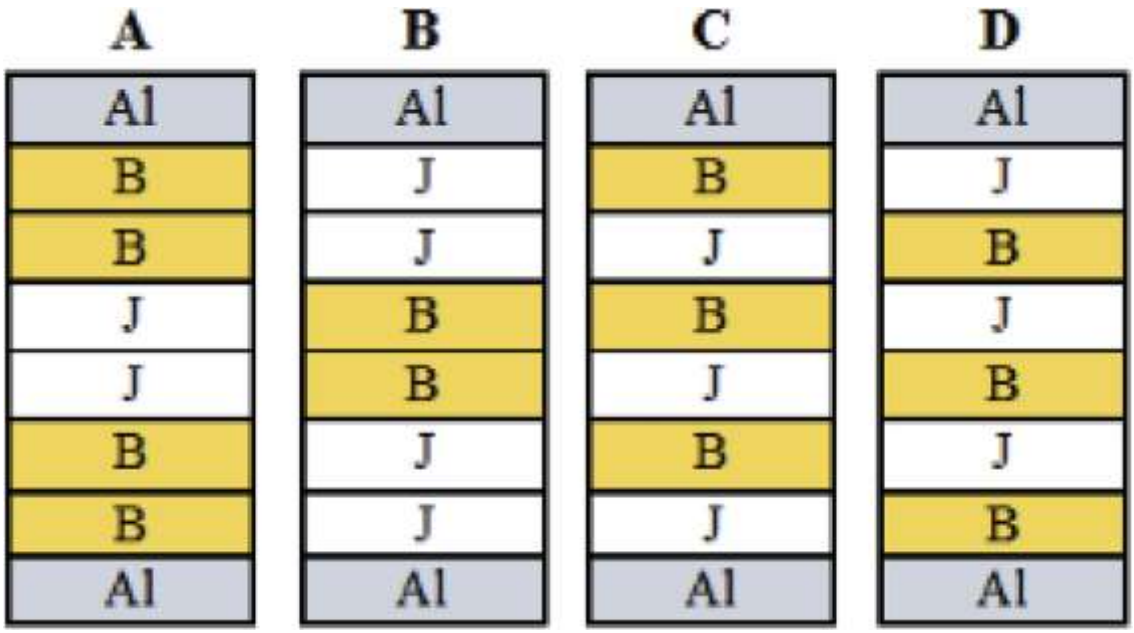

Fig. 1. The different stacking sequence of fiber metal laminates: A and B are the sandwich structures, and C and D are the intercalated structures. (Al: aluminium layer with thickness of 0.5 mm, B: basalt fibers with thickness of 0.32 mm, and J: jute fibers with thickness of 0.8 mm).

2.4. Mechanical testing

The tensile properties of FMLs were measured by a Hounsfield H25KS tester with the maximum load cell capacity of 25 kN. The tensile samples were cut and polished with the dimensions of 200 mm × 20 mm according to the ASTM D3039 standard, and the cross head speed of the tensile test was set to 2 mm/min. The interlaminar shear strength (ILSS) samples were cut and polished with the dimensions of 70 mm ×10 mm according to the ASTM D2344 standard. For more accuracy in each mechanical test, five samples were tested.

3. Results and discussion

3.1. Surface modification

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Fig. 2 shows the surface of untreated and treated jute fibers. It can be seen that the untreated jute fiber had a smooth surface, while the treated jute fiber exhibited a rather rough surface. The rougher surfaces of the natural fibers could help to improve the adhesion between the fibers and the matrix due to the interlocking with the matrix [27]. Saha et al. [28] also reported that NaOH treatment removed non-cellulosic materials like lignin, pectin or hemicellulose from an interfibriller region. Also, alkalization by NaOH led to fiber bundle fibrillation that caused a better contact to the matrix [16].

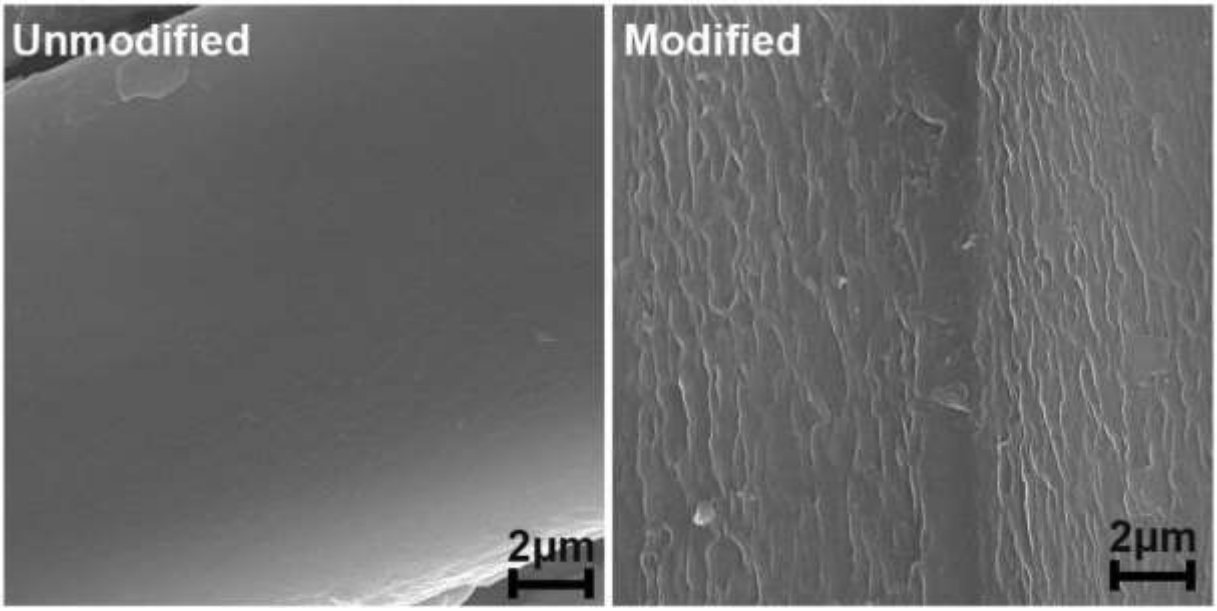

Fig. 2. The modified and unmodified surfaces of the jute fibers.

Fig. 3 shows the surface of the FPL treated aluminum sheet. The comparison of treated and untreated sheets showed that the treated sheet had the rougher surface along with micro-cavities. Based on the previous researches, the existence of these cavities and rough surfaces could encourage the epoxy matrix penetration into the aluminum sheets, creating the

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mechanical locks which would increase the adhesion between aluminum and the composite [29].

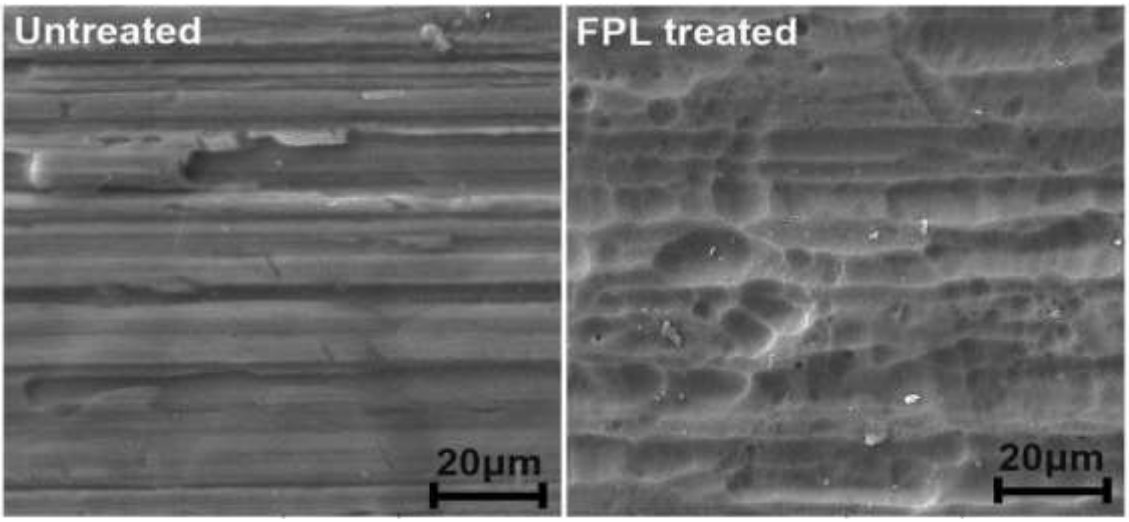

Fig. 3. The FPL of the treated and untreated aluminum layers.

3.2. Mechanical properties

The results obtained from tensile and ILSS tests are listed in Table 1. Fig. 4 shows the tensile strength data of the FMLs. Based on this figure, it could be seen that A and B sandwich structures had the highest and lowest tensile strength with the tensile strength of 213 and 139 MPa, respectively. It could also be realized that the tensile strength of C and D intercalated structures was very close. According to this figure, it could also be seen that the tensile strength of the A structure was approximately 53.2, 20.3 and 25.3% more than that of B, C and D structures, respectively. Also, the tensile strength of C and D structures was approximately 27.3% higher than that of the B structure.

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Table 1. The tensile and interlaminar shear strength results.

FML Structure	Tensile strength (MPa)	Elastic modulus (GPa)	Absorbed energy (kJ/m^3)	Strain (%)	Shear strength (MPa)
A	213 ± 6	39.5 ± 1.0	5.85 ± 0.2	5.46 ± 0.2	16.67 ± 0.3
B	139 ± 4	29.6 ± 0.8	6.06 ± 0.12	9.00 ± 0.3	10.30 ± 0.30
C	177 ± 5	34.3 ± 0.4	4.73 ± 0.1	4.93 ± 0.2	14.09 ± 0.20
D	170 ± 5	34.1 ± 0.4	4.69 ± 0.12	4.87 ± 0.1	13.92 ± 0.2

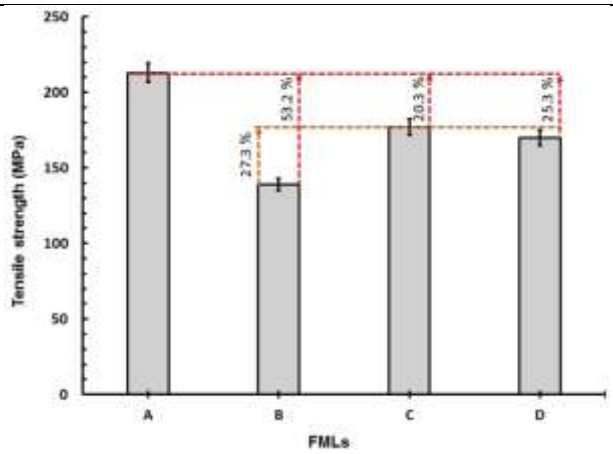

Fig. 4. The tensile strength of different FMLs structures.

Based on other literature [30,31], the outer layers sustain more load than the inner layers in the hybrid composites. So, if the stronger fibers are used as outer layers, the FML structures will have higher tensile strength. The other important factor is the adhesion between fibers and aluminum layers. The jute fibers have weak adhesion with the aluminum layers, due to the hydrophilic nature of them [32], whereas the basalt fibers have a cohesive adhesion with the aluminum layers [33]. So, the B and A structures had minimum and maximum tensile strength because of the mentioned factors.

Fig. 5 indicates the elastic modulus data of FML structures. According to this figure, A and B sandwich structures with the elastic modulus of 39.5 and 29.6 GPa had the highest and lowest elastic modulus, respectively, in comparison with other FML structures. Also, the C and

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D intercalated structures had a similar elastic modulus. Fig. 6 shows the tensile strain and the absorbed energy of the FML structures. Based on this figure, it could be seen that the B structure had maximum strain, in comparison with other structures. The trend in the absorbed energy values was similar to that of strain. As comparison with the basalt fibers, the jute fibers have two important features which are hollow structures and ability to fibrillations. As have been previously mentioned in other literature, the fibrillation and hollow structures can absorb the energy [34,35]. So, because of the highest weight fraction of the jute fibers, the B structures had the maximum energy absorption, in comparison with the other structures.

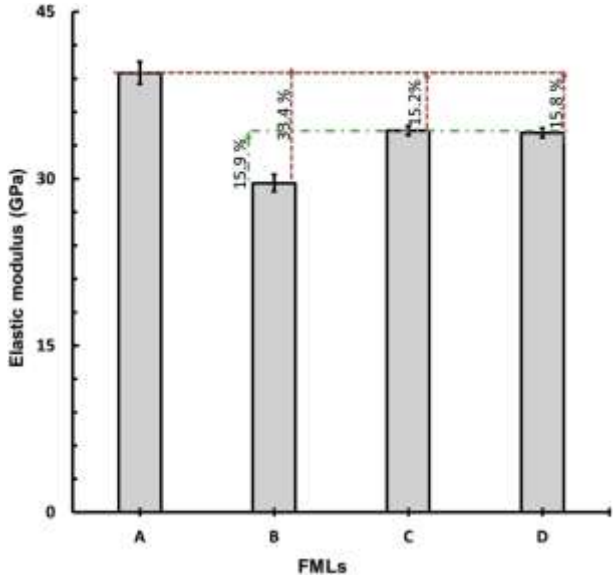

Fig. 5. The elastic modulus of different FMLs structures.

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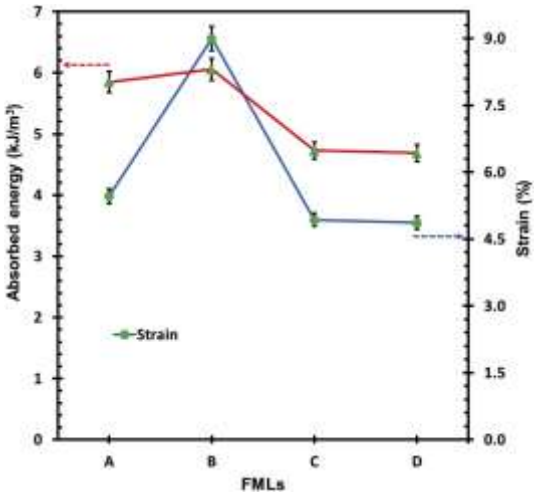

Fig. 6. The absorbed energy and the strain of different FMLs structures in the tensile test.

Fig. 7 shows the interlaminar shear strength (ILSS) of the FML structures. Similar to tensile strength, A and B sandwich structures with the ILSS of 16.67 and 10.3 MPa had the maximum and minimum ILSS values, respectively. The same was true for the C and D intercalated structures. Given Fig. 7, it could be seen that the ILSS of the A structure was approximately 61.8, 18.3 and 19.8% more than that of B, C and D structures, respectively. It could also be observed that ILSS in C and D structures showed the improvement of about 36.8% over the B structure.

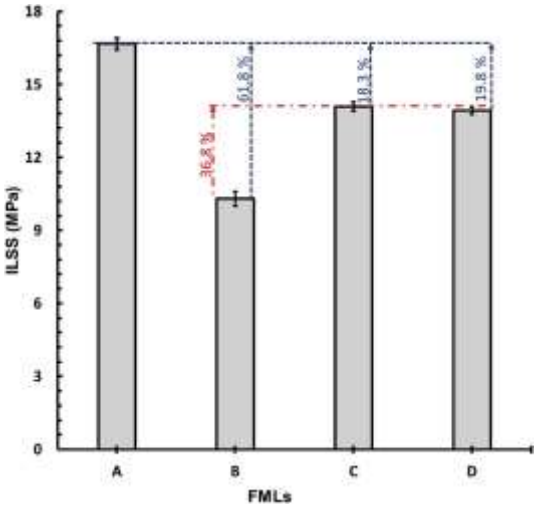

Fig. 7. The interlaminar shear strength of different FMLs structures.

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3.3. Macrostructural observations

Figs. 8-a to 8-d correspond to FMLs close-up views after the tensile test. From Fig. 8-a, it could be inferred that there was adequate adhesion between the aluminum sheet and the basalt fibers. Therefore, after the tensile test, some parts of the basalt fibers were adhered to the surface of the aluminum sheet. However, some delamination between the aluminum and basalt fibers could still be observed, while the delamination between the jute and basalt fibers was not evident. As can be seen, the basalt fibers were pulled out, whereas the jute fibers were fractured. Tearing of the aluminum sheets could be seen in these figures, but there was not sufficient evidence for the plastic deformation. Similar results have been observed by Vieira et al. [32]. Fig. 8-b exhibits a close-up view of the B structure. As can be seen, the adhesion between the aluminum and the jute fibers was too weak and the delamination between these layers was a dominant factor of failure. Similar results have been reported by Aghamohammadi et al. [33]. This was in contrast to the adhesion between the basalt and jute fibers, which was adequate. In this figure, like Fig. 8-a, the basalt fibers had been pulled out. Fig. 8-c and 8-d are the close-up views of C and D intercalated structures, respectively. The delamination between the aluminum and jute fibers, plastic deformation and tearing of aluminum sheets, the pull out of basalt fibers, and the failure of jute fibers were all phenomena which can be detected in these figures.

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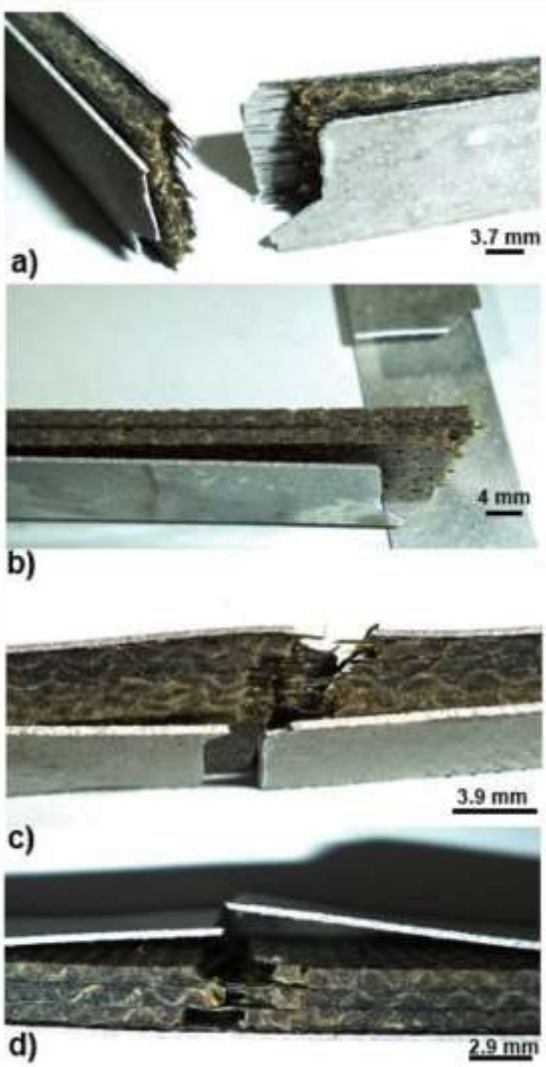

Fig. 8. The close-up view of different FMLs structures in the tensile test; a) A structure, b) B structure, c) C structure, and d) D structure.

Figs. 9-a to 9-d show the close-up views of A, B, C and D FML structures after the ILSS test. In Fig. 9-a, the plastic deformation could be observed in the front aluminum layer, whereas the delamination between the aluminum and the basalt fibers was shown in the back face of the FML structure. In the B FML structure, the delamination between the jute fibers and aluminum was the dominant failure mechanism. However, in the C structure, although delamination between the aluminum and the jute fibers was not evident, the plastic deformation of aluminum

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was observed. In the sample D, the delamination between the jute and the aluminum interface could not be observed. In this sample, the failure of the jute fibers and delamination between the basalt-jute fibers was the dominant failure mechanism due to the buckling phenomenon and the interface failure [36].

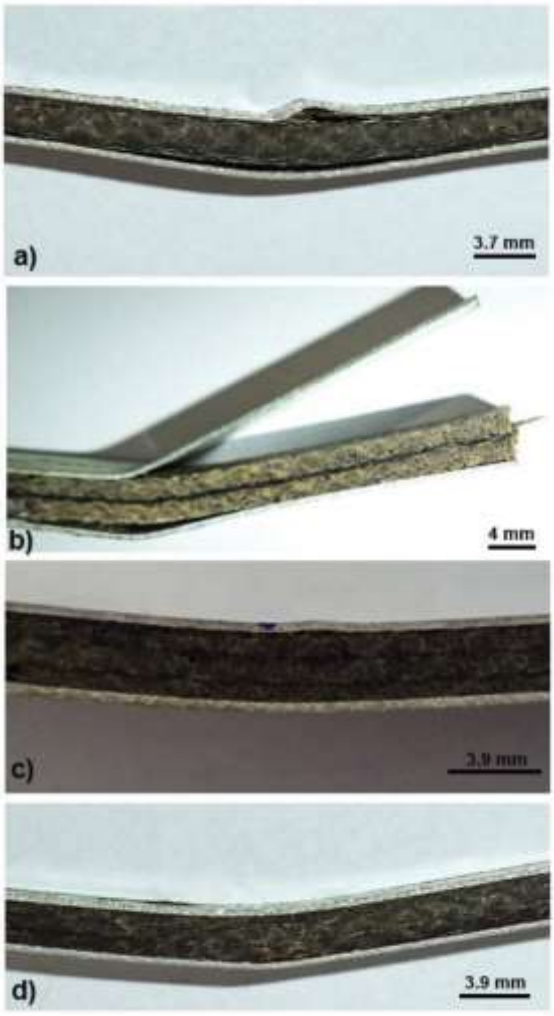

Fig. 9. The close-up view of different FMLs structures in the ILSS test; a) A structure, b) B structure, c) C structure, and d) D structure.

3.4. Microstructure

The observed differences in the behaviors of FML structures could be explained by microstructural analysis. Fig. 10 shows the microstructure of basalt and jute fibers interfaces

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with the aluminum sheet in the A and B structures, respectively. Based on Fig. 10-a, it could be seen that the interface of the basalt and aluminum sheet was very strong, so that some parts of the basalt fibers had stuck to the aluminum surface, although the pull out of the basalt fibers was detectable. The reason for this good adherence could be the diffusion of epoxy to the aluminum modified surface, creating mechanical locks [37]. As can be seen in Fig. 10-b, the jute fibers had no proper adhesion with the aluminum sheet, whereas some parts of the epoxy were stuck to the aluminum sheet. Fiore et al. [36] have reported that the jute fibers have weak adhesion with the epoxy matrix. Therefore, it could be concluded that the weak adhesion between the epoxy and the jute fibers was the main reason for the delamination between the jute fibers and the aluminum layer.

Fig. 11 shows the fractographs of the jute fibers in the A structure. In Fig. 11-a, the fibrillation, fibers pull out, and fibers fractures can be easily observed. Also, because of the fibers pull out, a huge hole was created by the initiation of secondary cracks. Based on Fig. 11-b, due to alkali treatment, the adhesion between the jute fibers and the epoxy was convincing. But the main issue was the adhesion between each fibril and the epoxy, which could mostly affect the transfer stresses from the matrix to fibrils. As can be seen in Fig. 11-C, the empty spaces existed in fibrils. These holes provided the best evidence showing that the epoxy could not wet the fibrils surfaces; therefore, it could not fill these holes. Therefore, due to this effect, stress transferring through the epoxy matrix was reduced; it even deteriorated the mechanical properties of the FML structures.

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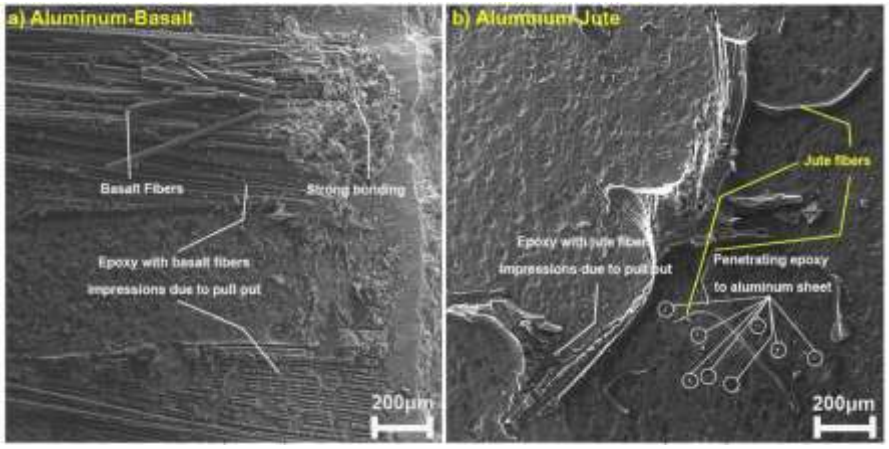

Fig. 10. The SEM observation; a) the interface between the basalt fibers and the aluminium layer, and b) the interface between the jute fibers and the aluminium layer.

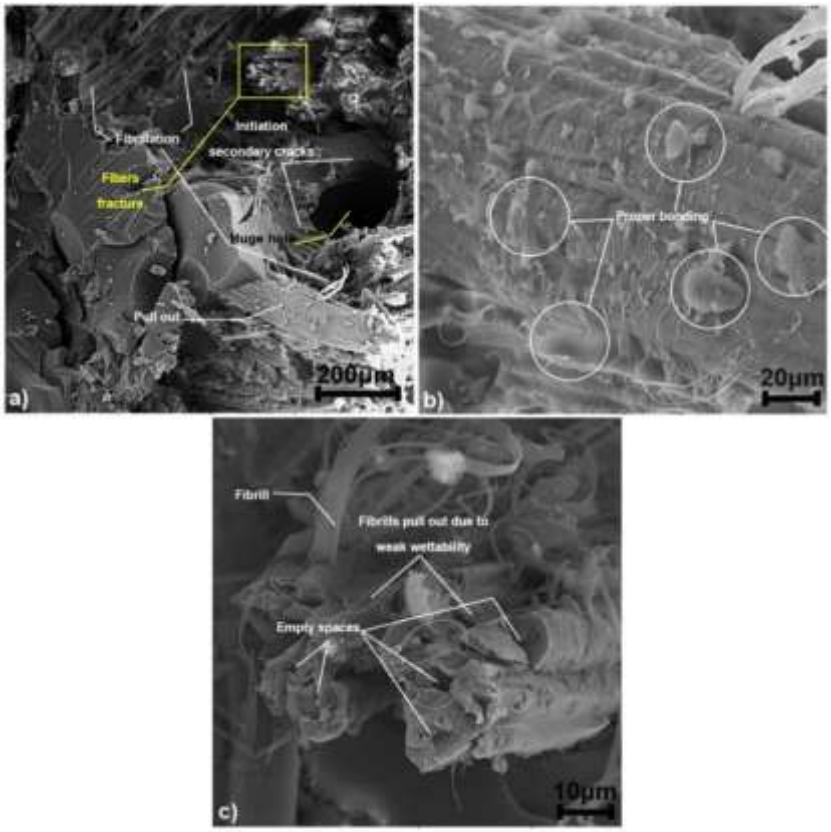

Fig. 11. The SEM observations in the interface between the jute fibers and the epoxy matrix in different magnifications: a) 150x, b) 1.00 kx, and c) 2.00 kx.

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Unlike the jute fibers, the basalt fibers in the A structure (Fig. 12) showed the strong bonding with the epoxy matrix and the matrix cracking was the dominant failure mechanism in this layer. This means that the stress transferring could be more readily achieved in the basalt fibers than in the jute fibers. Therefore, it could be argued that the significant difference between the mechanical properties in the sandwich structures A and B could be due to the difference in the ability of load transferring in the basalt and jute fibers.

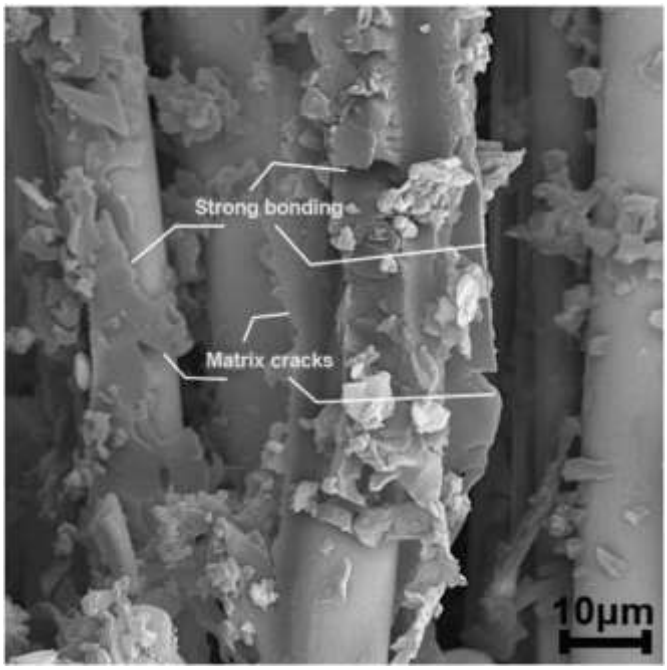

Fig. 12. SEM observation in the interface between the basalt fibers and the epoxy matrix.

4. Conclusions

In this work, the tensile properties and the interlaminar shear strength (ILSS) of environmentally-friendly fiber metal laminates (EFFMLs) fabricated by jute (J) and basalt (B) fibers as the hybrid reinforcement and aluminum (Al) 2024-T6 as the structure skin and the epoxy resin as the matrix were investigated. In this regard, two sandwiched structures of Al/J/J/B/B/J/J/Al and Al/B/B/J/J/B/B/Al and two intercalated structures of Al/J/B/J/B/J/B/Al

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and Al/B/J/B/J/B/ J/Al were fabricated. The following conclusions could be drawn from the present investigation.

1. The sandwich structure, in which the jute fibers had been sandwiched by the basalt fibers (Al/B/B/J/J/B/B/Al), had the highest tensile strength, elastic modulus and ILSS; these were 213 MPa, 39.5 GPa and 16.67 MPa, respectively.
2. The sandwich structure with the basalt fibres sandwiched by the jute fibers (Al/J/J/B/B/J/J/Al) had the lowest tensile strength, elastic modulus and ILSS; these were 139 MPa, 29.6 GPa and 10.3 MPa, respectively.
3. The (Al/J/J/B/B/J/J/Al) sandwich structure had the highest energy absorption, due to the fibrillation ability and hollow structures.
4. Both intercalated structures (Al/J/B/J/B/J/B/Al and Al/B/J/B/J/B/J/Al) had the similar tensile strength, elastic modulus and ILSS of approximately 34 MPa, 4.7 GPa and 14 MPa, respectively.
5. Macro and micro observation showed that the basalt fibers had the proper adhesion with the aluminium layers, whereas the jute fibers had the weak one with them.
6. Both basalt and jute fibers had the appropriate adhesion with the epoxy matrix, whereas the jute fibrils had the weak adhesion with it.
7. The macro and micro observations of the FML structures also revealed that the basalt fibers showed pull out and delamination from the aluminium sheet fracture behaviours, whereas the jute fibers exhibited the fibers fractures, pull out, delamination from aluminium sheet, and fibrillation fracture behaviours.

Appendix A. Supplementary data

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