

Effects of applying a combination of surface treatments on the mechanical behavior of basalt fiber metal laminates

M. Emami Mehr¹, H. Aghamohammadi¹, S. N. Hosseini Abbandanak¹

Gh. R. Aghamirzadeh², R. Eslami-Farsani^{1*}, S. M. H. Siadati¹

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

² Mechanical Engineering Department, Faculty of Engineering, South Tehran Branch, Islamic Azad University, Tehran, Iran

* Corresponding author: eslami@kntu.ac.ir

Abstract

The surface treatment of metals has a great role on the adhesion of the metals to the polymers. There are various surface treatment methods for adhesive joint applications. However, the effect of the combination of surface treatment methods on the mechanical behavior of adhesive joints has not been extensively studied. In this study, the effects of applying a combination of surface treatments on the flexural and Charpy impact behavior of fiber metal laminates (FMLs) were investigated. The surface treatments included forest product laboratory etching (FPL), sulfuric acid anodizing (SAA), sandblasting, sandblasting + FPL and sandblasting + SAA. The FMLs were made from Al2024-T3, basalt fibers and epoxy. Scanning electron microscopy (SEM) was used to study the surface morphology of the Al2024-T3 laminates and fracture surface of the samples. Furthermore, the surface roughness of the Al2024-T3 sheets after different surface treatments were evaluated using profilometry. Results showed that the adhesion of Al2024-T3 to polymeric layers was significantly affected by various surface treatments. Results of bending tests indicated that the highest bending strength and strain to failure values were respectively achieved for the SAA and sandblast treated samples. On the other hand, although there was a slight improvement in the bending strength, the application of SAA or FPL etching after sandblasting caused a negative effect on the strain to failure value of the samples. However, impact test findings showed that the combination of FPL or SAA treatments after sandblasting rendered positive effects on the low-velocity impact behavior of the FMLs.

Keywords: Fiber metal laminates; Adhesion; Surface treatments, Bending behavior, Impact behavior.

1. Introduction

Nowadays, hybrid composites especially fiber metal laminates (FMLs) have found widespread applications in aircraft structures. Due to the use of both metals and fiber reinforced polymer (FRP) layers in them, FMLs have superior properties over their constituents [1]. It is well known that the main causes of failure the aircraft components are due to the collision of foreign bodies with them, including the runway debris, dropped tools, bird strikes, high-velocity impacts, etc. [1, 2]. It has also been reported that FMLs used in aircraft are subjected to thermal cycling [3]. Furthermore, the most susceptible zone for the failure of FMLs is the metal-polymer interface [4]. Therefore, improving the metal-polymer bonding can be the most important factor for enhancing the reliability and durability of the FMLs.

Surface treatment of metals is one of the most significant key factors affecting the properties of metal-polymer bonds. Various surface treatment methods have been introduced for enhancing the adhesion of metals to polymers. Generally, these methods can be categorized into mechanical, chemical, electrochemical, coupling agents, etc. The use of surface treatment methods, prior to lamination, can significantly influence the surface morphology, surface energy and wettability of the metals and consequently the mechanical properties of FMLs [4]. In fact, the use of surface treatment methods can improve the mechanical properties of adhesive bonding through various mechanisms. The severe surface roughness of metals caused by mechanical methods leads to increasing the surface area for bonding and thus improving the adhesive bonding of FMLs [5]. On the other hand, there are chemical/electrochemical methods, such as changing the chemical

nature of the metal surface via the formation of an uniform oxide layer [6, 7]. The use of coupling agents results in the formation of covalent bonds, resulting in enhancing the bonding strength [4].

Over several decades, many researchers have examined the role of various surface treatments on the mechanical behavior of hybrid composites such as FMLs and single lap joints. However, there are few studies comparing the effects of various surface treatments on the morphology and roughness of the aluminum laminate and consequently, on the mechanical behavior of FMLs. In one of these studies, Gonzalez-Canche et al. [8] studied the effects of various surface treatments on the morphology, wettability and roughness of Al5052-H32 and their consequent effects on the interfacial adhesion of thermoplastic-based FMLs. Their results showed that the wettability and roughness of Al5052-H32 significantly increased due to the application of chemical-etching treatment as compared to other samples. Indeed, the shear strength of the chemically treated samples was higher than that for the sanded samples.

In another study, the effects of various surface treatments of abrasion, alkaline, Forest product laboratory (FPL) and P2 (Sulfo-Ferric) etch of Al1050 and Al2024 were investigated on the adhesion strength of single lap joints by Prolongo et al. [9]. They showed that in comparison to abrasion and alkaline methods, the use of P2 and FPL methods provided a better effect for enhancing the adhesion strength of single lap joints.

Different surface treatment methods have different mechanisms for improving the adhesion between metals and polymers. For example, increasing the surface area of the bonding is the main mechanism for enhancing the mechanical properties of surface treatments while creating a porous structure is the main mechanism of chemical/electrochemical methods. Therefore, it seems that due to the influence of different

This is the accepted manuscript (postprint) of the following article:

Mehr, M. E., Aghamohammadi, H., Abbandanak, S. H., Aghamirzadeh, G. R., Eslami-Farsani, R., & Siadati, S. M. H. (2019). Effects of applying a combination of surface treatments on the mechanical behavior of basalt fiber metal laminates. *International Journal of Adhesion and Adhesives*, 92, 133-141
<https://doi.org/10.1016/j.ijadhadh.2019.04.015>

mechanisms, the combination of surface treatments using different mechanisms cause the improvement of mechanical properties of FMLs. However, to the best of the authors' knowledge, fewer researchers studied the role of a combination of two or more surface treatments have on the mechanical behaviors of FMLs. In one study, Leena et al. [10] investigated the effects of aluminum surface treatments of solvent degreasing (SD), SD+FPL (SDFPL) and SDFPL + primer (SDFPLP) on the shear strength properties of the resulting adhesive bonds. Their results showed that the highest surface energy, surface roughness and lap shear strength were achieved for the SDFPL sample. Their results also revealed that although the use of primer after FPL-etching led to lowering of the contact angle, the surface roughness and total surface energy decreased. Therefore, the surface treatment SDFPLP was not appropriate for enhancing the lap shear strength of SDFPL treated sample. In another study, Laban and Mahdi [11] studied the effects of surface treatments of degreasing, alkaline-etching, O₂ plasma, N₂ plasma, laser and laser + N₂ plasma on the fracture toughness of aluminum/fiberglass epoxy reinforced laminates. Their results demonstrated that the use of alkaline etching treatment had superior effect on improving the fracture toughness of samples, compared to other treatments. However, they also reported that the combination of laser and N₂ plasma resulted in the highest improvement in the fracture toughness value.

It is well-known that glass, carbon and aramid fibers are the common fibers used in polymeric constituents of FMLs [1]. However, there are still few works based on fabrication of FMLs with natural fibers such as flax, hemp, kenaf, basalt, etc. [12, 13]. Basalt fibers have several outstanding properties such as high chemical stability, good resistance to high temperature, high mechanical strength, non-toxicity, non-combustibility, etc. [14].

The aim of this study was to investigate the effects of applying a combination of surface treatments of sandblasting with FPL and anodizing on the morphology and roughness of Al2024-T3. Furthermore, the effects of these surface treatments on the flexural and low velocity impact behaviors of FMLs made from Al2024-T3, basalt fibers and epoxy were investigated.

2. Experimental

2.1. Materials

Aluminum alloy grade 2024-T3 with 0.5 mm thickness, basalt fibers (BAS 350.1270.A, Basaltex, Belgium) and epoxy resin (Epon 828, with hardener TETA, supplied from Shimi Afsoon engineering materials company) were used for fabrication of samples. The resin to hardener ratio was 100:13 by weight, as recommended by the manufacturer.

2.2. Surface treatment of Al2024-T3

Before fabrication of FMLs, the various surface treatments including sulfuric acid anodizing (SAA), FPL, sandblast, sandblast + FPL and sandblast + SAA were performed on the surface of the Al2024-T3 alloy sheets. In the SAA method, the samples were first cleaned by acetone, then immersed in NaOH solution (0.2%) for 2 min and then immersed in distilled water for 10 min. Then, 5 ml HNO₃ (63%) was added to 500 ml distilled water and the samples were deoxidized and washed. Finally, the SAA process was done (0.5 molar, 6.65 A/m², 20 °C) for 10 min and then samples were washed with distilled water.

The chemical composition of the etching solution in the FPL method consisted of 33.7 g/l sodium dichromate (Na₂Cr₂O₇) and 374.5 g/l sulfuric acid, in accordance to ASTM D2674. The Al2024-T3 samples were first cleaned by acetone, then immersed in NaOH

solution (0.2%) for 2 min and etched for 30 min at 60 °C. After etching, the samples were immersed in distilled water for 5 min.

For sandblast treatment, SiO₂ particles (average size of 100 µm) were used. Fig. 1 shows the optical microscopy image of used SiO₂ particles in this study. Sandblasting was performed using a blaster which was connected to a high-pressure compressor which was located at an angle of 90°. During sandblasting, the distance between the nozzle (with the inner diameter of 8 mm) and the surface of A12024-T3 was kept at 40 cm and the process was continued for 3 min at the constant pressure of 0.01 MPa. Finally, the sheets were ultrasonically cleaned with acetone to remove any contamination.



Fig. 1. Optical microscopy image of used SiO₂ particles in sandblasting.

In sandblast + FPL and sandblast + SAA methods, at first the A12024-T3 sheets were sandblasted and then FPL and SAA treatments were performed on them.

2.3. Fabrication of basalt FMLs

Basalt FMLs with 2/1 configuration were fabricated by a hand lay-up method. The interlayer consisted of 4 layers of basalt fibers reinforced epoxy (BFRE). At first, the basalt fibers were impregnated with epoxy resin and then the A12024-T3 sheets were connected to the BFRE layers. It is important to note that the volume ratio of the resin to basalt fibers was 3:1. In addition, the volume ratio of the BFRE layers to aluminum sheets was 1:1. After laminating, the samples were placed in a mold for the curing step at 0.15 MPa and in ambient temperature. Applying pressure resulted in the reduction of bubbles and removal of excess resin. The samples were kept for 7 days at room temperature for complete curing process. Finally, for mechanical tests, the samples were cut into the required dimensions using a water-jet cutter.

2.4. Mechanical testing

Three-point bend test was conducted at ambient temperature according to ASTM D790 on samples of $150 \times 20 \times 1.9$ mm. The flexural strength and strain of the samples were calculated using equations (1) and (2), respectively.

$$\sigma_f = \frac{3PL}{2bd^2} \quad (1)$$

$$\varepsilon_f = \frac{6Dd}{L^2} \quad (2)$$

Where σ_f is the flexural strength (MPa), ε_f is the flexural strain, P is the maximum load (N), L is the support span (mm), b is the width of sample (mm), d is the thickness (mm) and D is the maximum deflection at the center of the sample (mm).

In addition, low-velocity impact behavior of samples was investigated using Charpy impact tests. Charpy impact tests (without crack) were performed on the samples of $90 \times 10 \times 1.9$ mm. The Charpy impact tests were performed under constant impact energy of

This is the accepted manuscript (postprint) of the following article:

Mehr, M. E., Aghamohammadi, H., Abbandanak, S. H., Aghamirzadeh, G. R., Eslami-Farsani, R., & Siadati, S. M. H. (2019). Effects of applying a combination of surface treatments on the mechanical behavior of basalt fiber metal laminates. *International Journal of Adhesion and Adhesives*, 92, 133-141 <https://doi.org/10.1016/j.ijadhadh.2019.04.015>

20 J according to the ASTM D6110 standard. Specific absorbed energy (S.A.E.) of samples was measured according to equation (3):

$$S.A.E = \frac{E_1 - E_2}{M.A} \quad (3)$$

Where E_1 and E_2 are the initial and final potential energy (J), respectively. M is the weight of the sample (g) and A is the area of the sample (cm^2).

2.5. Structural analyses

A Surtronic + 3 profilometer according to DIN EN IS 0274:1998 was used to characterize the effects of surface treatment methods on the roughness of the Al2024-T3 sheets. Prior to roughness testing, samples were kept in sonication bath and acetone for 20 min. For each specimen, five measurements at different locations were performed using a cut-off length of 0.8 mm and an evaluation length of 4 mm and finally, the average values were reported. The morphology of the used SiO_2 particles was evaluated by optical microscope (Meiji Techno OM). Moreover, the surface morphology of Al2024-T3 sheets after various surface treatments and the fracture surface of FMLs were analyzed using scanning electron microscopy (SEM). The analysis was accomplished using Quant FEG 250 SEM. In addition,

3. Results and discussion

3.1. Surface morphology of Al2024-T3

The surface morphologies of Al2024-T3 sheets after various surface treatments are presented in Figs. 2-6. As observed in Fig. 2, the surface morphology of Al2024-T3 after FPL-etching mainly consists of microscale pits uniformly distributed on its surface. On the other hand, the surface morphology of Al2024-T3 after SAA mainly consists of

microscale pits and nanoscale pores (Fig. 3). Clearly, the sizes of microscale pits on anodized Al2024-T3 surface are much lower than those for FPL-etched samples. On other hand, the uniformity of microscale pits on the FPL-etched samples is higher than that of SAA-treated samples. It is important to note that the after FPL etching or SAA treatment, a porous structure of alumina (Al_2O_3) is formed on the surface of aluminum. The formed alumina layer has nanoscale pores. On other hand, the appearance of microscale pits can be attributed to the presence of Al_2Cu intermetallic particles in Al2024-T3 [15]. According to the difference between the chemical activity of aluminum and Al_2Cu , it can be said that galvanic corrosion has occurred for the anodized and FPL-etched samples. Thereby, comparing the size of microscale pits in the FPL and anodized samples revealed that FPL-etching caused higher galvanic corrosion as compared to SAA.

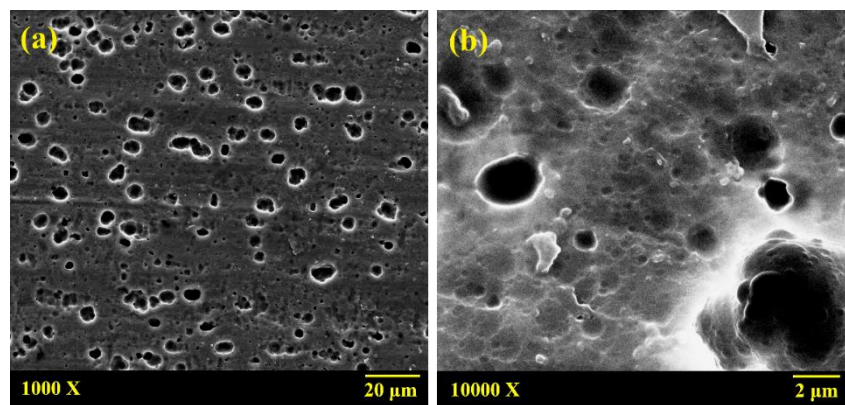


Fig. 2. The surface morphology of Al2024-T3 sheets after FPL-etching at different magnifications.

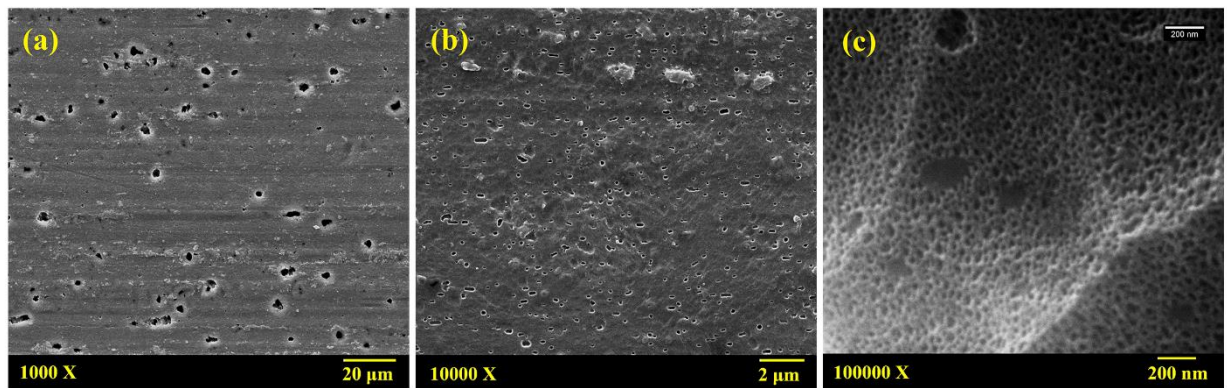


Fig. 3. The surface morphology of Al2024-T3 sheets after SAA at different magnifications.

Fig. 4 shows SEM micrographs of A12024-T3 surfaces after sandblasting at different magnifications. The high roughness with high humps and valleys of the surface is clearly visible. In addition, Figs. 5 and 6 show the surface morphologies of sandblast + FPL and sandblast + SAA treated samples, respectively. It is clear that after etching/anodizing treatment the sharp edges of sandblasted samples disappeared. This phenomenon can be attributed to the fact that sharp edges have high energy (activity) and accordingly dissolved in chemical solutions. In addition, the presence of microscale pits in sandblast + FPL treated sample is more visible than sandblast + SAA treated one. In other words, it can be said that there is a combination of peaks and valleys (due to sandblasting) and microscale pits for sandblast + FPL/SAA treated samples. Furthermore, the surface morphology of sandblast + FPL treated A12024-T3 is much rougher than sandblast + SAA treated A12024-T3. This observation can be explained by more aggressive galvanic corrosion of the FPL treatment.

This is the accepted manuscript (postprint) of the following article:

Mehr, M. E., Aghamohammadi, H., Abbandanak, S. H., Aghamirzadeh, G. R., Eslami-Farsani, R., & Siadati, S. M. H. (2019). Effects of applying a combination of surface treatments on the mechanical behavior of basalt fiber metal laminates. *International Journal of Adhesion and Adhesives*, 92, 133-141
<https://doi.org/10.1016/j.ijadhadh.2019.04.015>

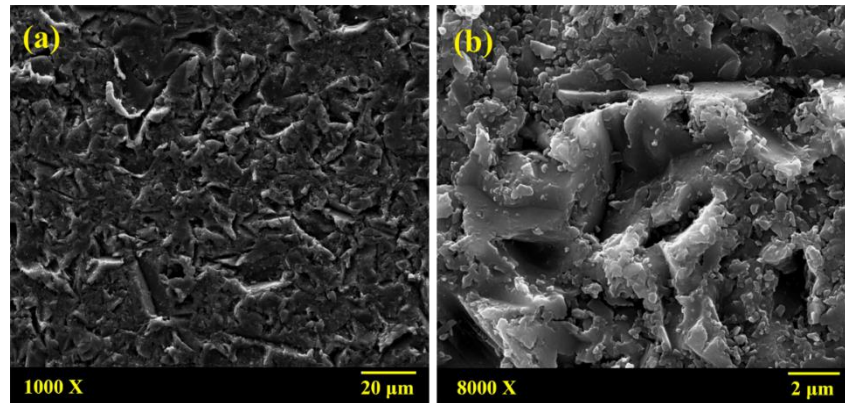


Fig. 4. The surface morphology of Al2024-T3 sheets after sandblasting at different magnifications.

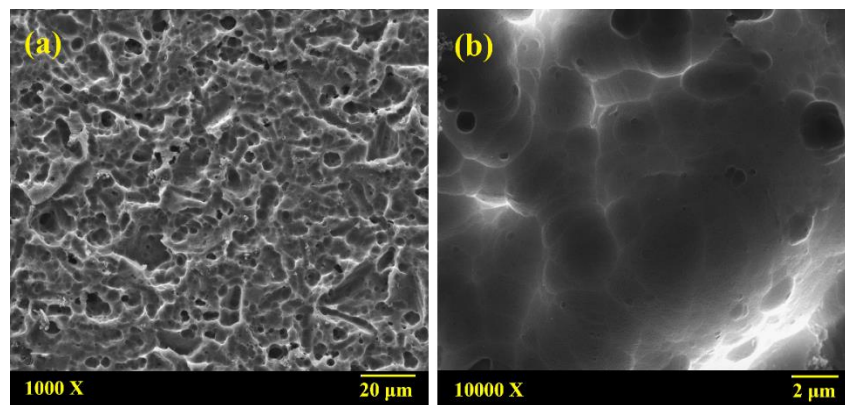


Fig. 5. The surface morphology of Al2024-T3 sheets after sandblast + FPL at different magnifications.

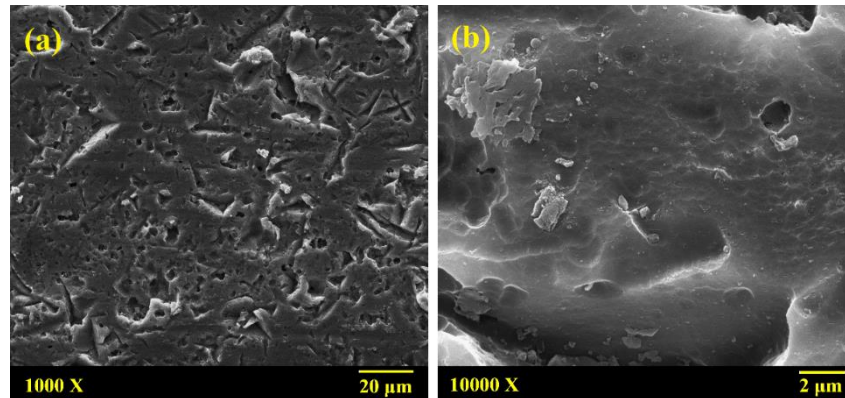


Fig. 6. The surface morphology of Al2024-T3 sheets after sandblast + SAA at different magnifications.

3.2. Profilometry results

Fig. 7 shows the surface roughness value of Al2024-T3 sample after various surface treatment methods obtained by profilometry. It can be said that the SAA and FPL-etching methods led to the creation of a relatively smooth surface compared to the sandblasting method. In other words, the roughness value of the sandblasted Al2024-T3 is about 1.06 μm which is respectively 4.2 and 2.9 times higher than those of SAA and FPL treated Al2024-T3. Furthermore, it seems that the SAA and FPL treatments after sandblasting had a significant effect on increasing the roughness value of Al2024-T3 surface. In this regard, among all samples, the sandblast + FPL sample showed the highest roughness value of 1.63 μm . The higher surface roughness of sandblast + FPL compared to sandblast + SAA sample can be due to the higher microscale pits on it, increasing peak to valley distance.

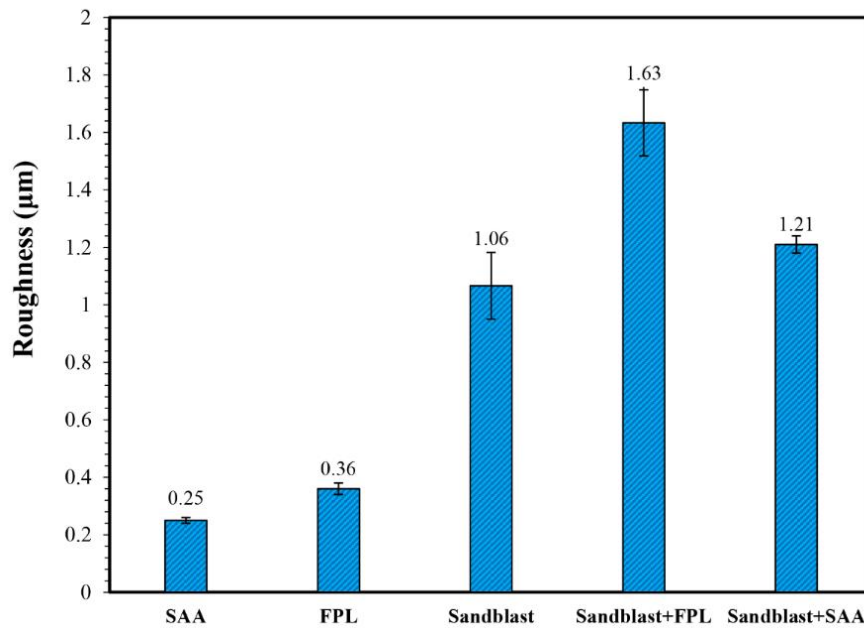


Fig. 7. Surface roughness of A12024-T3 after various surface treatments.

3.3. Flexural testing

After bending tests, the surface of all samples was not damaged and no delamination was occurred between laminates. The results from 3-point bending tests are shown in Fig. 8. It is clear that there are no significant differences between the flexural strengths of SAA and FPL treated samples. However, the flexural strength of sandblasted samples (637.40 MPa) is about 3.4% lower than that for SAA treated samples (659.29 MPa). On other hand, it can be seen that FPL-etching and SAA followed by sandblasting has resulted in increasing the flexural strength of sandblasted samples; the flexural strength of sandblast + SAA treated samples (655.33 MPa) was about 2.8% higher than that of sandblasted samples. From these results, it can be deduced that although the presence of microscale pits is useful for improving the adhesion strength, their large sizes can weak the mechanical properties of aluminum sheets. Thereby, the higher flexural strength of SAA

treated samples can be attributed to the presences of smaller pits on the surface of aluminum compared to FPL treated samples.

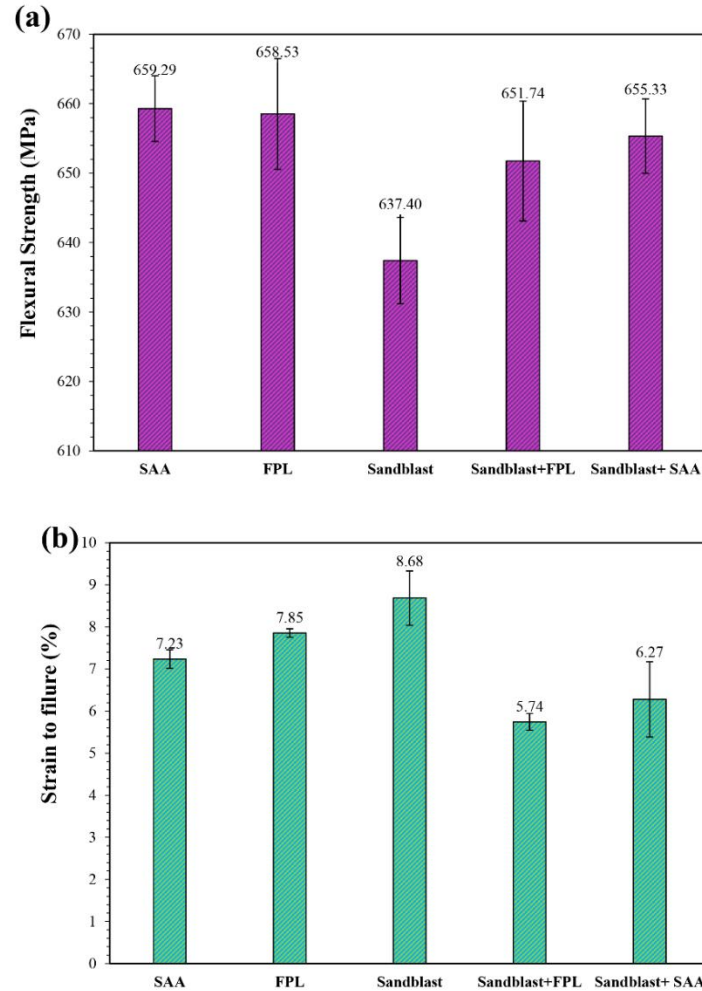


Fig. 8. Three-point bend test results (a) flexural strength and (b) strain to failure.

In addition, the strain to failure values of different samples are given in Fig. 8-b. Contrary to the flexural strength values, the highest strain to failure value of about 0.0868 was achieved for sandblasted sample which is about 20.05% higher than that for SAA treated sample. Contrary to what was mentioned about the role of FPL/SAA treatment after sandblasting, these treatments caused remarkable decrement of strain to failure value

of sandblasted sample. In other words, the strain to failure values of sandblasted sample decreased respectively about 38.43% and 51.2% after SAA and FPL-etching. Therefore, it can be concluded that FPL/SAA treatments after sandblasting, in spite of the little improvement in the flexural strength, had detrimental effects on the flexural properties of FMLs.

Presentation of SEM images of the fracture surfaces is necessary to understand the reasons for the aforementioned results. The cross-section of FPL, SAA and sandblast treated samples are presented in Fig. 9. For all samples, there were good adhesion and mechanical interlocking between A12024-T3 and polymeric layers. However, there were no visible differences between the cross-sections of samples. The SEM images of A12024-T3 fracture surfaces of different samples are depicted in Figs. 10-14. It is well established that the adhesive (interfacial) and cohesive (interlaminar) failures are the main types of failure in hybrid composites. Adhesive failure is referred to the failure at the metal/polymer interface while cohesive one is related to the failure within polymeric layers.

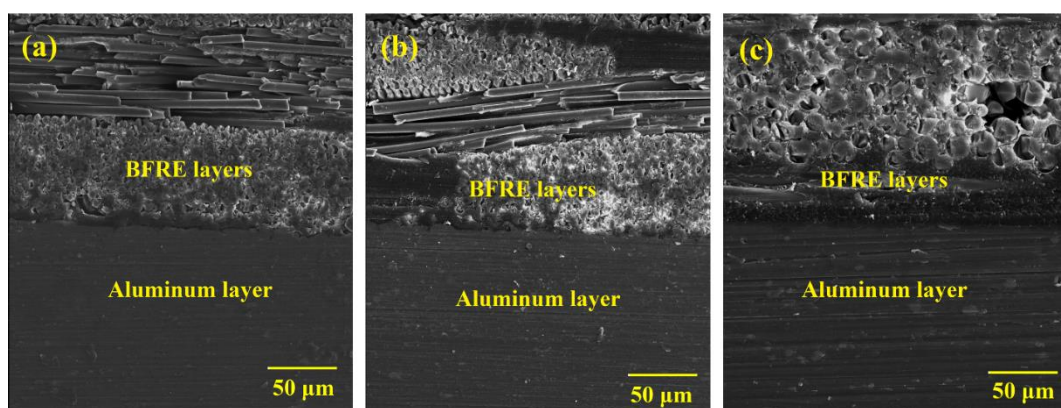


Fig. 9. Cross- section of (a) FPL, (b) SAA and (c) sandblast FMLs.

Close observation of the fracture surfaces indicate that a mixture of adhesive and cohesive failures has occurred in all samples. It is clear that for FPL and sandblasted samples a small amount of BFRE layers has remained on the A12024-T3 surface (Fig. 10), indicating high contribution of adhesive failure rather than cohesive failure. However, the large amount of BFRE layers in fracture surface of SAA samples reveals the high contribution of cohesive failure. Accordingly, it can be concluded that the adhesion of A12024-T3 to epoxy for SAA samples is in better condition than for FPL and sandblasted samples. These results indicate the better mechanical performance of SAA treated samples compared to FPL treated ones.

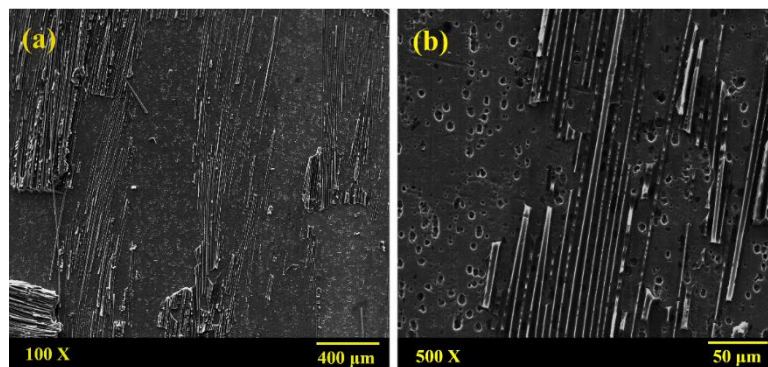


Fig. 10. SEM image of A12024-T3 fracture surface in FPL treated FMLs at different magnifications.

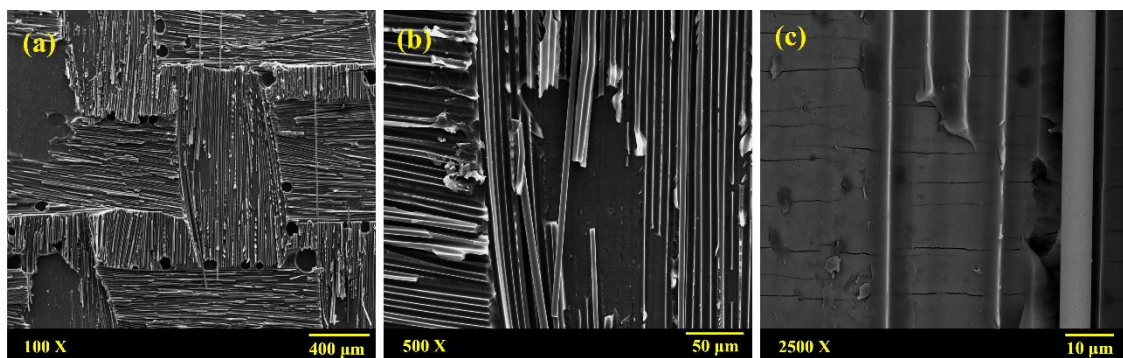


Fig. 11. SEM image of A12024-T3 fracture surface in SAA treated FMLs at different magnifications.

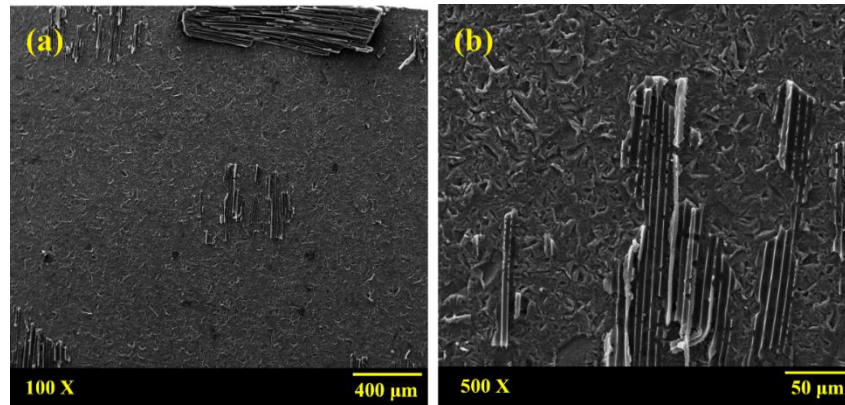


Fig. 12. SEM image of A12024-T3 fracture surface in sandblast treated FMLs at different magnifications.

Figs. 13 and 14 show the A12024-T3 fracture surface of sandblast + FPL and sandblast + SAA treated samples, respectively. It is clearly visible that there are no significant differences between the amount of BFRE debris on the surface of sandblast and sandblast + FPL treated samples, indicating that FPL was not effective in increasing the contribution of the cohesive failure. However, the remained BFRE layers on the surface of sandblasted A12024-T3 are significantly larger after the application of SAA treatment. In addition, the SEM images of A12024-T3 fracture surface of different samples at higher magnification (Fig. 14-c) revealed that the application of SAA after sandblasting had a great role in increasing the mechanical interlocking mechanism rather than the FPL treatment. Therefore, it can be concluded that the adhesion of A12024-T3 to epoxy improved considerably upon using SAA treatment.

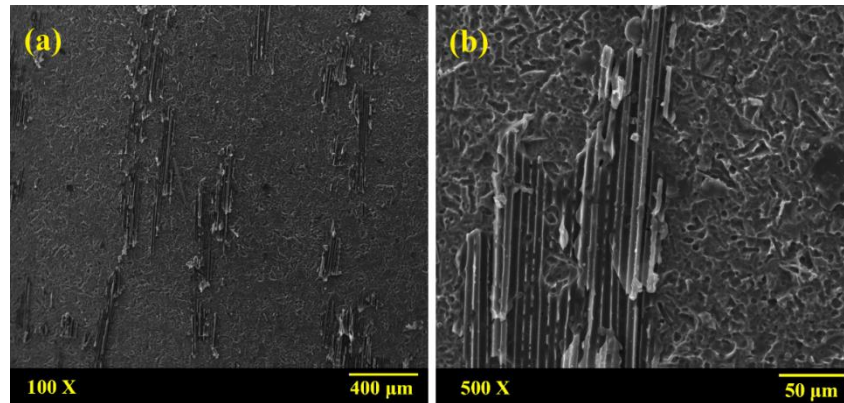


Fig. 13. SEM image of A12024-T3 fracture surface in sandblast + FPL treated FMLs at different magnifications.

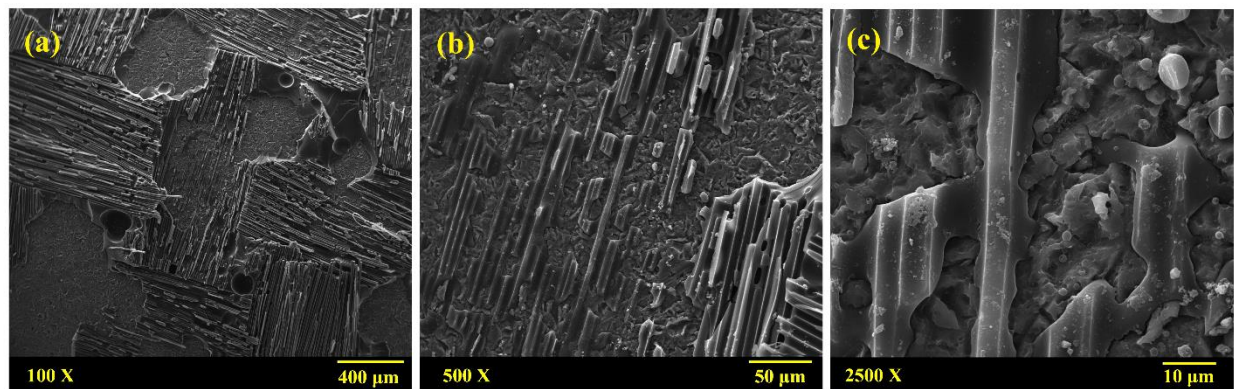


Fig. 14. SEM image of A12024-T3 fracture surface in sandblast + SAA treated FMLs at different magnifications.

In contrast to the SEM results, however, strain to failure values of sandblasted samples were much higher than those for all samples. Moreover, as mentioned earlier, the roughness value of sandblasted samples increased due to the use of SAA or FPL treatments. It is well-known that the higher surface roughness provides more surface area for adhesive bonding. Therefore, it is expected that the sandblast + FPL/SAA samples show better mechanical performance than other samples. However, contrary to the SEM

observations and roughness results, as mentioned above, the flexural properties (strain to failure) of sandblast + FPL/SAA decreased as compared to sandblasted samples.

Regarding the abovementioned results, it is necessary to present the possible reasons for the inconsistency between the SEM results and mechanical behavior of the samples. It is important to note that, in flexural testing, the occurrence of cracking in the bottom side (tension side) of the metal sheet is the main reason for the failure of all samples. Fig. 15 shows the macroscopic images of sandblast + FPL treated samples after bending tests. In Fig. 15a, the high deflection of samples after bending tests is visible, demonstrating the proper adhesion between the laminates. In addition, in Fig. 15b the cracking in the bottom side of aluminum is visible. The surface morphology of the aluminum sheets greatly influence on the occurrence of these cracks.

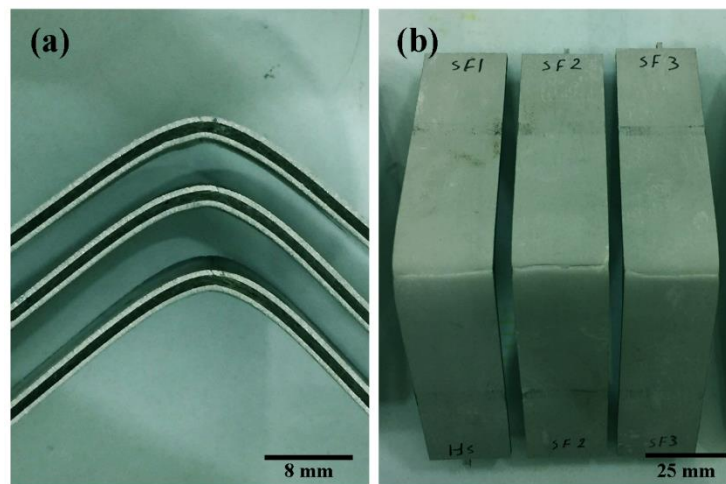


Fig. 15. The macroscopic images of sandblast + FPL treated samples after bending tests from two sides.

The microscale pits created by FPL or SAA treatments are the susceptible sites for crack initiation. Fig. 11-c, clearly shows that the cracks in the A12024-T3 layer. Close observation in Fig. 11-c also reveals that just about all cracks passed through the pits. As

This is the accepted manuscript (postprint) of the following article:

Mehr, M. E., Aghamohammadi, H., Abbandanak, S. H., Aghamirzadeh, G. R., Eslami-Farsani, R., & Siadati, S. M. H. (2019). Effects of applying a combination of surface treatments on the mechanical behavior of basalt fiber metal laminates. *International Journal of Adhesion and Adhesives*, 92, 133-141
<https://doi.org/10.1016/j.ijadhadh.2019.04.015>

mentioned earlier, the presence of microscale pits on the surface of FPL and SAA treated A12024-T3 is related to galvanic corrosion between aluminum and Al_2Cu intermetallic particles. At the same time, it has been reported that pits are the most crack initiation sites in aluminum alloys [16, 17]. Therefore, the lower strain to failure value of SAA and FPL treated samples compared to sandblasted ones can be related to the presence of these pits. For example, the fatigue behavior of A17075 alloy with different surface treatments of pickling and chromic acid anodizing was studied by Shahzad et al. [16]. They showed that the anodizing and pickling treatments respectively led to 43% and 31% reduction of fatigue strength as compared to the untreated samples. Their observations demonstrated that pits were the most initiation sites for fatigue crack propagation. In another similar study, Aghamohammadi et al. [18] investigated the role of anodizing time on the flexural properties of basalt FMLs. Their results showed that with increasing the anodizing time, the size of microscale pits increased and consequently the flexural strength and toughness values decreased.

Moreover, as it is well known, performing the sandblast treatment leads to the generation of compressive residual stress. In the literature, there are considerable works on the effects of compressive residual stresses on the mechanical behavior of aluminum alloys. For example, Mhaede [19] investigated the effect of ball-burnishing (BB) and shot peening (SP) on the fatigue properties of A17075-T73. He reported that the use of SP resulted in the higher surface roughness of aluminum, in comparison with BB. His results showed that the fatigue strength of aluminum improved because of the use of BB and SP surface treatments. It has been reported that increasing the surface roughness and compressive residual stress caused by various surface treatments such as sandblasting, SP, BB, etc. have opposite influence on the fatigue behavior of aluminum. In fact, higher

surface roughness leads to early crack nucleation while higher residual compressive stress causes the reduction of crack growth rate. Therefore, there should be a competition between the performance of roughness and compressive residual stresses. Thereby, the improvement in strain to failure of sandblasted samples over SAA and FPL treated samples is due to the compressive residual stresses caused by sandblasting.

The other remarkable result is related to the lower strain to failure of sandblast + FPL and sandblast + SAA treated samples. It was mentioned earlier that the surface roughness of sandblasted samples increased owing to the use of FPL or SAA treatments. Moreover, in these samples the presence of microscale pits is visible. Therefore, although the compressive residual stresses are beneficial in delaying the crack growth, it can be concluded that the higher surface roughness along with microscale pits have the synergetic effect of decreasing the strain to failure of these samples.

3.4. Charpy impact testing

The S.A.E. values of different samples after Charpy impact testing are given in Fig. 16. Similar to flexural strength values, the sandblasted sample had the lowest S.A.E. value among all samples. In this regard, the S.A.E. value of sandblasted sample ($16.99 \text{ J.cm}^2/\text{g}$) is about 17.83% lower than that for SAA sample ($20.02 \text{ J.cm}^2/\text{g}$). However, it can be seen that the lower S.A.E. value of sandblasted sample is compensated using FPL/SAA treatments after sandblast; the S.A.E. value of sandblast + SAA sample ($20.61 \text{ J.cm}^2/\text{g}$) is about 2.94% higher than that for SAA samples. Therefore, it can be deduced that the combination of FPL/SAA treatments with sandblasting has positive effect on the low-velocity impact behavior of FMLs.

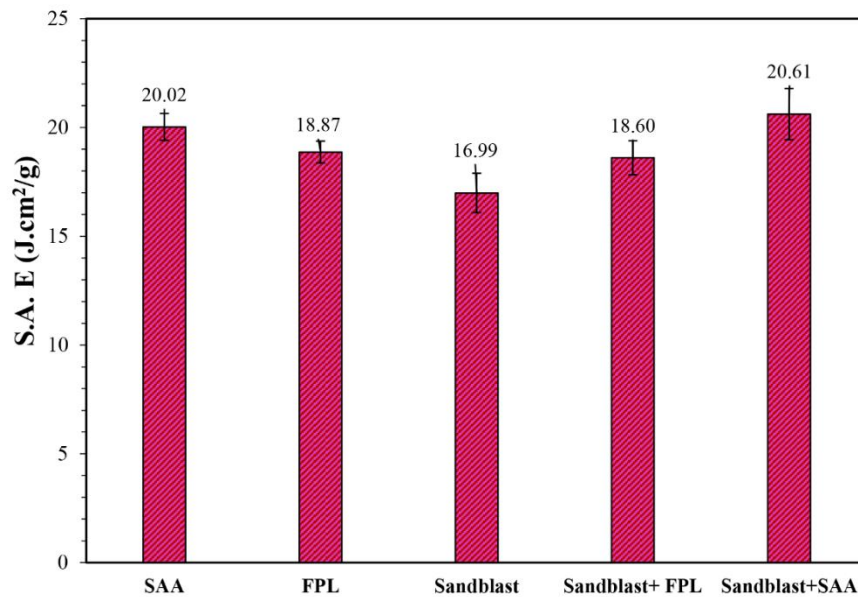


Fig. 16. The S.A.E. value of different samples from Charpy impact test.

The results of flexural and Charpy impact tests revealed that there should be different mechanisms affecting the flexural and impact behaviors of FMLs. It is well-known that fiber bridging and delamination are the main mechanisms for energy absorption during impact testing. Delamination (debonding) in the adjacent layers of FMLs causes delaying in failure of fibers in the delaminated regions. Also, fiber bridging leads to load transfer from the metal layer to the stronger fibers in the adjacent FRP layers. Fiber bridging is a phenomenon which occurs after delamination. However, excessive delamination/debonding leads to the failure of the structure [20]. In this regard, due to its effect on debonding, the adhesion of metal to polymeric layers has a significant role on the impact response of FMLs. Accordingly, in impact testing, the adhesion of metal/polymer interface is more important than in flexural testing. On other hand, as observed in the SEM images, the adhesion of A12024-T3 to epoxy improved in sandblast + SAA and sandblast + FPL treated samples as compared to sandblast sample. In other words,

improvement in the adhesion of A12024-T3 to epoxy in sandblast + SAA and sandblast + FPL treated samples is the main reason for the achieved improvement in impact testing. In addition, regarding the importance of impact behavior, it can be deduced that the sandblast + SAA sample is the most suitable sample for aircraft components.

4. Conclusions

In this study, the surface treatments of sulfuric acid anodizing (SAA), Forest product laboratory (FPL), sandblast, sandblast + FPL and sandblast + SAA on A12024-T3 sheets were applied and their surface morphology and roughness were studied using SEM observations and profilometry, respectively. Fiber metal laminates (FMLs) consisting of A12024-T3 sheets and basalt fiber reinforced epoxy layers were fabricated via hand lay-up method. The main aim of this study was to investigate the effects of a combination of two or more surface treatments on the flexural and Charpy impact properties of FMLs; combined application of sandblasting with FPL etching and SAA. Therefore, the following items were the main conclusions:

- 1- The use of FPL-etching and SAA treatments led to the formation of surfaces with microscale pits while the sandblast treatment caused the generation of a rough surface containing peaks and valleys. In addition, the combination of sandblasting with SAA or FPL-etching caused severe high surface roughness.
- 2- Profilometry indicated that the highest surface roughness was achieved for the sandblast + FPL treated A12024-T3 followed by the sandblast + SAA treated one.
- 3- SEM observations showed that the best adhesion of A12024-T3 to epoxy was achieved for the SAA treated sample. Moreover, the adhesion of the sandblasted

A12024-T3 to epoxy improved due to the application of SAA (or FPL-etching) and was comparable with the SAA treated FMLs.

- 4- Among all samples, the highest and lowest flexural strength values were achieved for SAA and sandblast treated FMLs, respectively. However, the highest strain to failure value of about 0.0868 was achieved for the sandblast treated sample, which was about 20.05% higher than that for SAA treated sample. On the other hand, the strain to failure value of sandblasted sample decreased by about 38.43% and 51.2% after SAA and FPL-etching, respectively.
- 5- The results of impact testing showed that the S.A.E. value of sandblasted sample was the lowest among the samples and was about 17.83% lower than that for the SAA sample. On the other hand, S.A.E. value of sandblast + SAA samples was about 2.94% higher than that for SAA sample. In summary, it can be concluded that the best impact behavior was achieved for the sandblast + SAA treated sample and this sample, among all samples, can be the best candidate for aircraft applications.

5. Future work

Further experimental studies are needed to investigate the effect of corrosive environments on the durability of the FMLs with different surface treatments.

References

- [1] Sinmazçelik T, Avcu E, Bora MÖ, Çoban O. A review: Fibre metal laminates, background, bonding types and applied test methods. *Materials & Design*. 2011;32(7):3671-85.

This is the accepted manuscript (postprint) of the following article:

Mehr, M. E., Aghamohammadi, H., Abbandanak, S. H., Aghamirzadeh, G. R., Eslami-Farsani, R., & Siadati, S. M. H. (2019). Effects of applying a combination of surface treatments on the mechanical behavior of basalt fiber metal laminates. *International Journal of Adhesion and Adhesives*, 92, 133-141
<https://doi.org/10.1016/j.ijadhadh.2019.04.015>

- [2] Sadighi M, Alderliesten RC, Benedictus R. Impact resistance of fiber-metal laminates: A review. *International Journal of Impact Engineering*. 2012;49:77-90.
- [3] Da Costa AA, Da Silva DF, Travessa DN, Botelho EC. The effect of thermal cycles on the mechanical properties of fiber–metal laminates. *Materials and Design*. 2012;42:434-40.
- [4] Park SY, Choi WJ, Choi HS, Kwon H, Kim SH. Recent Trends in Surface Treatment Technologies for Airframe Adhesive Bonding Processing: A Review (1995–2008). *The Journal of Adhesion*. 2010;86:192-221.
- [5] Harris A, Beevers A. The effects of grit-blasting on surface properties for adhesion. *International Journal of Adhesion and Adhesives*. 1999;19(6):445-52.
- [6] Mohamad M, Marzuki HFA, Bakar SNA, Abdullah AN, Ubaidillah EAE, Abidin MFZ, Omar S. Effect of anodizing electrolyte for structural adhesive bonding study of aluminum-carbon laminate composites. *International Journal of Automotive and Mechanical Engineering*. 2014;10:2091-101.
- [7] Xu Y, Li H, Shen Y, Liu S, Wang W, Tao J. Improvement of adhesion performance between aluminum alloy sheet and epoxy based on anodizing technique. *International Journal of Adhesion and Adhesives*. 2016;70:74-80.
- [8] Gonzalez-Canche N, Flores-Johnson E, Cortes P, Carrillo J. Evaluation of surface treatments on 5052-H32 aluminum alloy for enhancing the interfacial adhesion of thermoplastic-based fiber metal laminates. *International Journal of Adhesion and Adhesives*. 2018; 82:90-99.
- [9] Prolongo S, Ureña A. Effect of surface pre-treatment on the adhesive strength of epoxy–aluminium joints. *International Journal of Adhesion and Adhesives*. 2009;29(1):23-31.
- [10] Leena K KKA, Bhuvaneswari S, Suraj S, LakshmanaRao V. Effect of surface pre-treatment on surface characteristics and adhesive bond strength of aluminium alloy. *International Journal of Adhesion & Adhesives*. 2016;70:265-70.

- [11] Laban O, Mahdi E. Enhancing mode I inter-laminar fracture toughness of aluminum/fiberglass fiber-metal laminates by combining surface pre-treatments. *International Journal of Adhesion and Adhesives*. 2017;78:234-9.
- [12] Daghigh V, Khalili S, Farsani RE. Creep behavior of basalt fiber-metal laminate composites. *Composites Part B: Engineering*. 2016;91:275-82.
- [13] Vieira LMG, dos Santos JC, Panzera TH, Rubio JCC, Scarpa F, Novel fibre metal laminate sandwich composite structure with sisal woven core, *Industrial crops and products* 99 (2017) 189-95.
- [14] Sim J, Park C, Characteristics of basalt fiber as a strengthening material for concrete structures, *Composites Part B: Engineering* 36(6-7) (2005) 504-12.
- [15] Fin N, Dodiuk H, Yaniv A, Drori L. Oxide treatments of Al 2024 for adhesive bonding-surface characterization. *Applied surface science*. 1987;28(1):11-33.
- [16] Shahzad M, Chieragatti R, Mabru C, Rezai-Aria F, Surface characterization and influence of anodizing process on fatigue life of Al 7050 alloy, *Materials & Design* 32(6) (2011) 3328-35.
- [17] Zupanc UJG. Effect of pitting corrosion on fatigue performance of shot-peened aluminium alloy 7075-T651. *Journal of Materials Processing Technology*. 2010;210:1197-202.
- [18] Aghamohammadi H, Abbandanak SNH, Eslami-Farsani R, Siadati SH, Effects of various aluminum surface treatments on the basalt fiber metal laminates interlaminar adhesion, *International Journal of Adhesion and Adhesives*. 2018; 84:184-93.
- [19] Mhaede M. Influence of surface treatments on surface layer properties, fatigue and corrosion fatigue performance of AA7075 T73. *Materials and Design*. 2012;41:61-6.
- [20] Pärnänen T, Kanerva M, Sarlin E, Saarela O. Debonding and impact damage in stainless steel fibre metal laminates prior to metal fracture. *Composite Structures*. 2015;119:777-86.