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Aluminum surface treatment for shear strength improvement of the laminate composites

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Abstract. In this paper, the effect of surface treatment on the adhesion between D16AM aluminum alloy and thermoplastic polyurethane is studied. Adhesion between the metal and polymer plays a key role in the formation of the strength properties of the metal-polymer based laminar composites, which are nowadays widely used in load-bearing structures like beams, columns, roofs, pedestrian bridges, etc. The effects of the metal surface treatment, such as chemical etching, electrochemical anodizing, laser treatment, and a combination of the mentioned processing, on the composite's shear strength were examined. The morphology of the metal surface after treatment was studied via scanning electron microscopy and atomic force microscopy. The true value of the adhesive strength was calculated, taking into account the micro- and macro-relief of the surface and its actual surface area determined via atomic force microscopy. A combined method of metal surface treatment to increase adhesion to a polymer is proposed, the method includes chemical or electrochemical etching followed by laser treatment. It was shown that complex treatment of the aluminum surface allows to increase the shear strength of composites by 50 %. It is established that with chemical etching or laser treatment, the value of the true adhesive strength remains virtually unchanged, however, when using the combined method, its significant increase is observed.

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1. *Introduction*

Metal-polymer based laminar composites (MPLCs), consisting of alternating layers of metal and polymer, belong to the class of lightweight structural materials [1,2]. These materials find applications in load-bearing structures like beams, columns, roofs, multifunctional panels, pedestrian bridges, etc., contributing to improved construction practices and efficiency. Due to combination of light weight, sufficiently high strength, low cost, and easy technological process, MPLCs are widely used in government supported public infrastructure upgrade, thus, it is predicted that the construction composites market will exceed $65 billion by 2025 [3].

Laminar composites consist of continuous two-dimensional panels or sheets aligned for maximum high-strength, their qualities influenced by the constituent materials' characteristics and the geometrical design of the structural elements [1]. The main constituents in MPLCs are layers of metal, usually lightweight ones, for example, aluminum or titanium, and fiber-reinforced polymer. The layered structure of the MPLC provides a significant reduction in the rate of crack growth [4]. When a crack initiates and propagates in one of the layers at the interface, its growth stops until a crack appears in the next layer, and so on. MPLCs dissipate and dampen vibrations via a similar mechanism, and, thus, can be used as damping structural materials [5-7].

The main parameters affecting the properties of MPLCs are the thickness of the layers and their number, the properties of the constituents itself, the quality of pre-preg impregnation, the metal sheet surface condition, and interlayer adhesion. Adhesion between the metal and polymer plays a key role in the formation of the strength properties. It is known [8-10] that adhesion can be increased by increasing the metal layer surface roughness, since this leads both to an increase in the actual metal-polymer contact area and to the engagement of the liquid polymer to the unevenness of the metal surface.

Surface treatment is usually used for metal substrate preparation providing removal of contamination, good wettability, high roughness, mechanical and chemical stability [11,12]. It was shown that a number of factors are important for the adhesion: a stable oxide on the surface is important as this can prevent or minimize the formation of a relatively weakly bound inorganic layer; topography, which favours mechanical keying and provides an increased area over which interfacial interactions can occur is clearly beneficial. There are many methods of metal surface treatment [13]; among chemical ones, the most promising are etching in iron sulfate [14], nitric acid [15], sulfuric acid anodizing [16], and anodizing in a mixture of sulfuric acid with aluminum sulfate. In addition to chemical methods, physical methods are also widely used, the most popular are sandblasting [17,18]. It was found that the chemical treatment resulted in a more wettable and deeper roughness surface than the achieved using a sanded and degreased treatment and the chemical treatment yielded a superior cleaned surface, which in junction with the roughness area, resulted in a superior interfacial contact between the metal and the adhesive-composite material [15,19].

Recently, it was also shown that the laser processing [20-25] offers a fast, easy, and clean operation in metal microstructuring and can be a new strategy to improve joint strength. Laser cleaning enabled to increase dramatically the adhesion between the substrate irrespective of the previous aluminium surface condition [19]. It was shown [20] that laser texturing improved AA7475 surface roughness and wettability and also confirmed that the Al-O-C chemical bond represents the secondary bonding mechanism along the micro-mechanical interlocking. The results presented in the paper [21] have indicated that the laser micropatterning of AISI 430 stainless steel samples have a marked influence on the surface roughness and surface grove morphology, and consequently on joining properties of the materials.

In this paper, the effect of surface treatment of aluminum sheets on the shear strength of the MPLC was investigated. This paper aims to understand how laser treatments affect the mechanical behaviour of joints and the possible improvement achieved by adopting the sequence of laser and chemical treatment steps. Testing the shear strength and studying the surface morphology were conducted.

2. Methods

To manufacture the composites, layers of aluminum alloy D16AM of 0.5 mm thick (hereinafter referred to as aluminum) and thermoplastic polyurethane (TPU) film (OOO NPF Vitur) of 0.1 mm thick were used.

The aluminum samples were pre-cleaned with detergents, etched in a 10 % NaOH solution, and then washed with water. The exception was a series of samples processed by laser at different fill factors (D), in which the aluminum samples were only wiped dry, without cleaning agents.

Three types of treatment were carried out: 1) chemical etching in solutions of nitric acid and ferrous sulfate; 2) electrochemical treatment in electrolytes based on sulfuric acid and aluminum sulfate; 3) and laser treatment. The treatment parameters are given in Table 1.

Table 1. Aluminum surface treatment parameters.

№	Treatment type	Designation in the text	Processing details
1	Chemical	HNO_3	HNO_3 – 32 %; T = 20 °C; t = 1 min
2		$Fe_2(SO_4)_3$	$Fe_2(SO_4)_3$ 127 g/l; H_2SO_4 185 ml/l; T = 65 °C; t = 8 min
3	Electrochemical	H_2SO_4	H_2SO_4 200 ml/l; T = 20 °C; t = 20 min;
4		$Al_2(SO_4)_3$	$Al_2(SO_4)_3$ 200 g/l; H_2SO_4 60 ml/l; T = 20 °C; t = 20 min; j = 1.5 A/dm^2
5	Physical	Laser	P = 10 Вт; λ = 1 μm; D = 50 %; t = 5 ns; T = 20 °C; V = 1 m/s

The composites were manufactured by hot pressing. Two plates of size of 80×10 mm were placed in a mold with an overlap of 10 mm. One layer of TPU was placed between the plates. Pressing temperature was 200 °C, time was 20 min.

The shear strength test was performed according to GOST R 57066-2016 on a Zwick//Roell Z050 machine with a constant deformation rate of 1 mm/min. Surface microstructure studies were carried out using a Phenom Pro X scanning electron microscope. Atomic force microscopy (AFM) studies were conducted for a more thorough analysis of the surface relief. Basing on the preliminary study of the optimal scanning parameters, areas of 40x40 μm were analyzed since this provides high data reliability but does not lead to a radical increase in scanning time.

3. Results and Discussion

Fig. 1 shows the microscopic images of the aluminum surfaces after different types of etching. The surface treated in the HNO_3 solution has minimal porosity (Fig. 1a). Maximum porosity was observed after chemical etching in the $Fe_2(SO_4)_3$ (Fig. 1b). The pores' size does not exceed 5 μm. Surfaces treated electrochemically in sulfuric acid and aluminum sulfate electrolytes are almost identical (Figs. 1c, 1d). It should be noted that the pores formed during chemical etching (Figs. 1a, 1b) are shallower than those formed during electrochemical treatment (Figs. 1c, 1d).

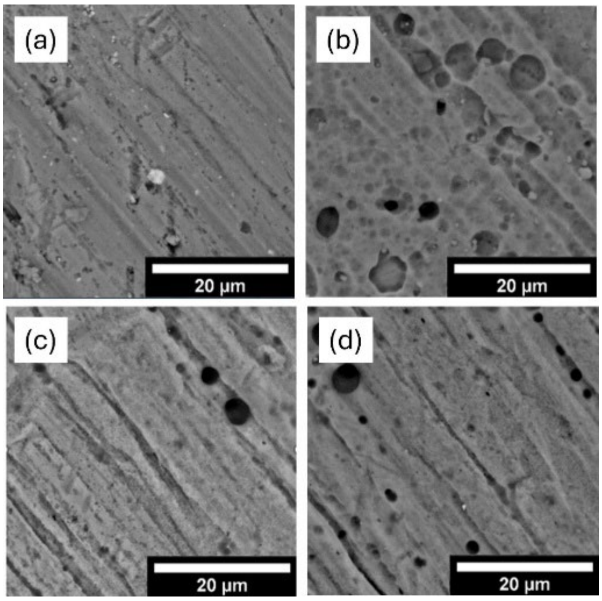

Figure 1. Surface morphology of aluminum depending on the etching:
a – HNO_3; b – $Fe_2(SO_4)_3$; c – H_2SO_4; d – $Al_2(SO4)_3$.

The results of shear strength tests depending on the aluminum surface etching type are presented in Fig. 2 (orange bars). The shear strength of the composite generally correlates with the observed porosity of the metal surface. Low roughness of the surfaces treated in HNO_3 solution resulted in a lower strength. Etching in $Fe_2(SO_4)_3$, H_2SO_4, $Al_2(SO_4)_3$ showed fairly close values of shear strength (5±0.5 MPa). However, the shear strength values of the specimens after treatment in $Fe_2(SO_4)_3$ have a much wider spread of values revealing inhomogeneity of the surface condition. This can be explained by the difficulty of maintaining the same concentration of the etchant during the treatment. Since during etching at an elevated temperature (65 °C), there is active evaporation of the components, which changes the ratio of the etchant components. The main advantages of the chemical etching in $Fe_2(SO_4)_3$ are simplicity, high efficiency, and high productivity. The disadvantage of the method is low environmental safety, since the vapors formed during etching are chemically active. The advantages of electrochemical processing are high repeatability, high quality, higher shear strength of the MPLC, and higher environmental friendliness. However, unlike chemical etching, anodizing is a more difficult method to implement, as it requires an adjustable power source and cooling, since the efficiency of the process decreases with increasing temperature.

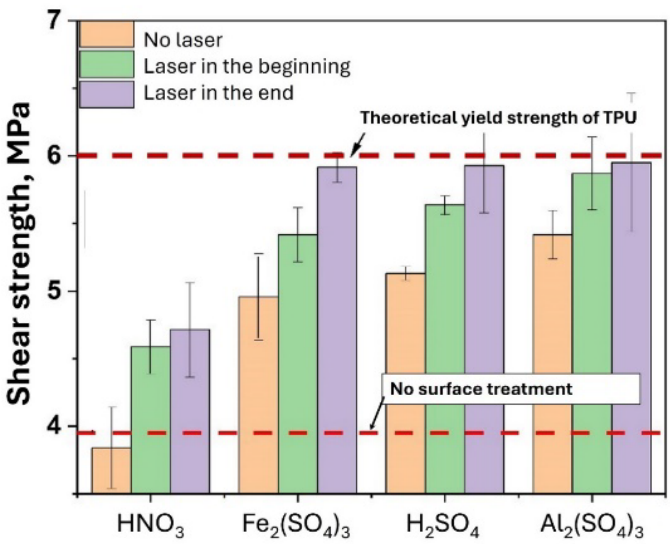

Figure 2. Shear strength of the composites depending on the aluminum surface treatment.

Microphotographs of the aluminum surface in the initial state and after laser treatment with fill factors *D* = 50 % are shown in Fig. 3. After laser treatment, there is a distinctive structure – craters formed due to the expulsion of molten metal from the heating zone.

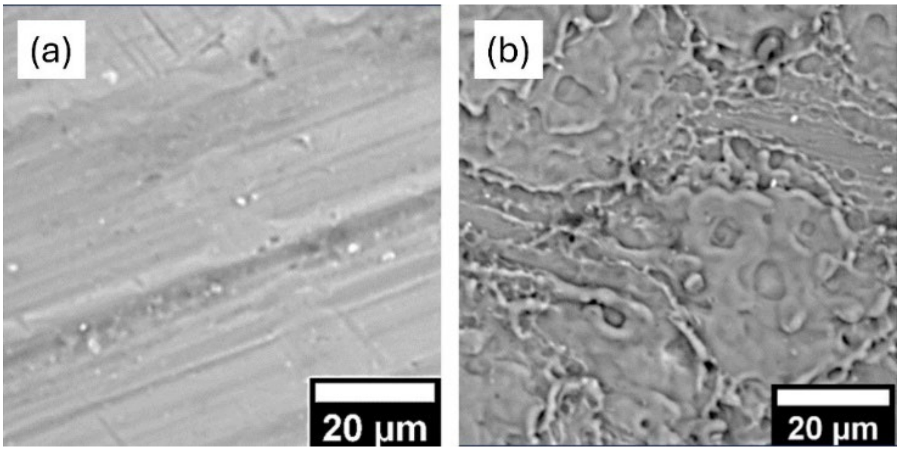

Figure 3. Surface morphology of aluminum:
a – initial state; b – after laser treatment with *D* = 50 %.

Next, the effect of combination of etching and laser processing and their sequence on the surface condition and shear strength of the composite was studied.

Fig. 2 shows the results of the complex surface treatment on the shear strength of the composite. As seen, laser treatment is effective both before and after chemical or electrochemical treatment. However, a greater effect appears if the laser treatment is carried out after any etching. Thus, changing the order between etching in $Fe_2(SO_4)_3$ and laser treatment allowed to increase the shear strength by 10 %. Regardless of the preliminary treatment, after laser treatment, all samples (except those treated in HNO_3) acquire the same shear strength, because the maximum shear strength of the composite is limited to the yield strength of TPU, which is about 6 MPa.

The effect of laser treatment is most noticeable after etching in $Fe_2(SO_4)_3$. In this case, the increase in shear strength was about 20 %. The morphology of the aluminum surface after different modes of complex treatment is practically identical: the surface is covered with "splash" spots as a result of laser treatment (Fig. 4a). An exception is the treatment mode in which chemical etching in iron sulfate $Fe_2(SO_4)$ was carried out after laser treatment (Fig. 4b). Instead of "splash" spots, the surface has developed porosity, significantly exceeding the porosity acquired after chemical etching of the surface not treated with a laser (Fig. 1b).

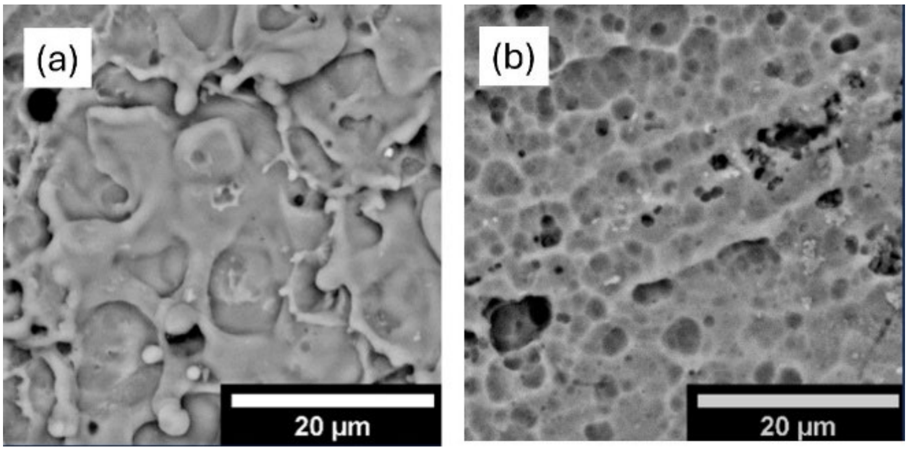

Figure 4. Surface morphology of aluminum depending on the sequency of etching in $Fe_2(SO_4)_3$ and laser processing: a – $Fe_2(SO_4)_3$ →Laser; b – Laser→$Fe_2(SO_4)_3$.

The visually observed surface relief obtained because of various complex treatments does not show a strict correlation with the shear strength of the composite: the samples with visually identical surfaces demonstrate different level of share strength. Based on this, it can be assumed that the difference in strength is due to roughness at the submicron level or the chemical composition of the surface layer (for example, the presence or absence of an oxide film, functional groups, etc.). For a more thorough analysis of the surface relief, surfaces analyses using AFM were carried out. AFM images of the aluminum surface in the initial state and after various processing modes are shown in Fig. 5.

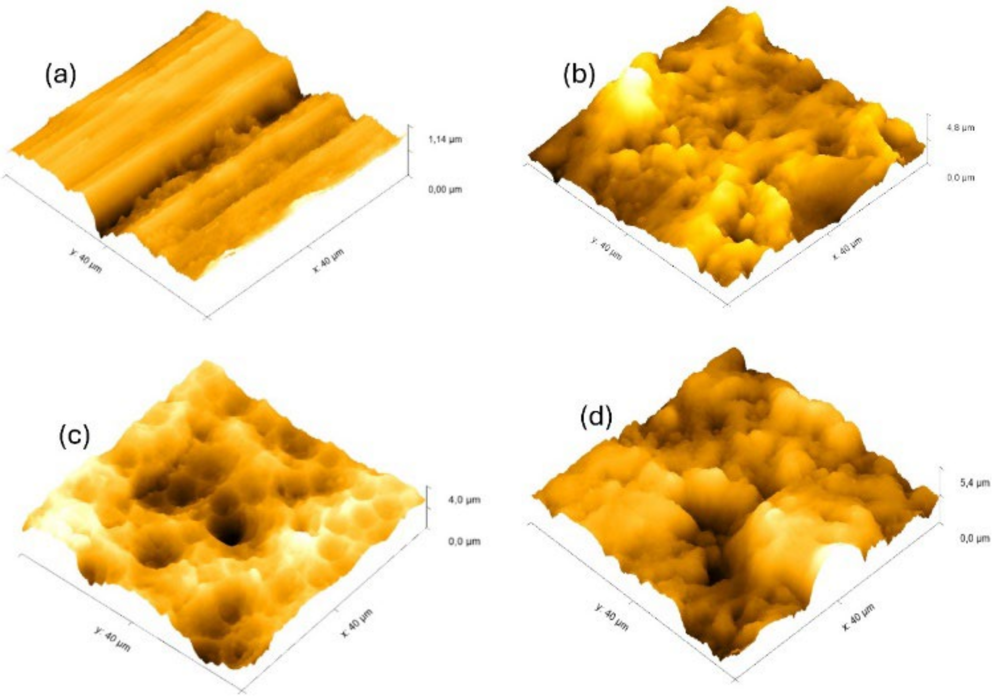

Figure 5. AFM images of the aluminum surface in initial state (a); after laser treatment (b); after complex treatment: laser followed by $Fe_2(SO_4)_3$ (c) and vice versa $Fe_2(SO_4)_3$ followed by laser (d).

Results of AFM measuring of the actual surface area $\left(S_f\right)$ are presented in Table 2. The ratios of the projected surface area $\left(S_0\right)$ to the actual one $\left(S_f\right)$ are presented in the form of a dimensionless normalization coefficient $\left(S_n\right)$:

$$S_n = \frac{S_f}{S_o}. \quad (1)$$

The data obtained show that all types of processing provide an increase in the actual surface area from 14 to 21 %. The maximum increase in the surface area was reached via complex processing –

chemical etching in $Fe_2(SO_4)_3$ followed by laser processing. Next, the true shear strength $\left(\tau_{true}\right)$ was calculated as the ratio of the measured shear strength $\left(\tau_{sh}\right)$ to the normalization coefficient $\left(S_n\right)$:

$$\tau_{true} = \frac{\tau_{sh}}{S_n}. \tag{2}$$

The calculation results are given in Table 2.

Table 2. Surface characteristics of aluminum and shear strength of the joint depending on the processing method.

Surface treatment	S_0, μм²	S_f, μм²	S_n	τ_{sh}, MPa	τ_{true}, MPa	$\frac{\tau_{true}}{\tau_0}$
Without treatment	1600	1660	1.04	3.9	3.7 (τ_0)	–
$Fe_2(SO_4)_3$		1820	1.14	4.7	4.1	**1.10**
Laser		1876	1.17	4.4	3.7	**1.00**
Laser→$Fe_2(SO_4)_3$		1835	1.15	5.4	4.7	**1.27**
$Fe_2(SO_4)_3$→Laser		1933	1.21	5.9	4.9	**1.32**

It was found that when using only one type of processing (etching or laser processing), the ratio of τ_{true} to $\tau_0 \left(\frac{\tau_{true}}{\tau_0}\right)$ has a value close to 1. This suggests that the observed increase in shear strength is associated only with the increase in the actual area of the surfaces being joined. However, when combining two processing methods, a significant increase in true shear strength, more than 25 % $\left(\frac{\tau_{true}}{\tau_0} = 1.27 \div 1.32\right)$, is observed. This allows us to conclude that during the complex processing, a significant change in the chemical composition of the thin surface layer of aluminum occurs, which has a great impact on the adhesive strength of the joint and on the final properties of the composite.

It is already known that the enhancing the adhesion between dissimilar materials represents a key aspect of producing structural hybrid multi-material structures [19]. Additional chemical or laser treatment of the samples surface before hot pressing of metal-polymer composites leads to an increase in shear strength by approximately 25 %. It was shown that the laser treatment of sample surface significantly increase actual surface area of Al-based materials as compare with just chemical treatment of the samples, however, true shear strength remains about the same even with the samples without any treatment, and that means the absence of any additional chemical bonding for the laser treated surface and polymer.

Combined chemical and laser treatment of the sampled provides further increase in shear strength of the material by almost 50 % compared to the original samples without treatment and gives significant increase of true stress as compare with simple single treatment of Al surface.

As a result of the AFM study, it was established that complex treatment of the aluminum surface, including both etching and laser treatment, leads to a significant increase in adhesion to thermoplastic polyurethane, while the mechanism of influence of such treatment consists not only in the increase in the actual surface area but also in its chemical modification. That shows the best results in as compare with modern study of plastic-Al composites (+39 % strength increase with the two-step laser texturing + cleaning treatment), R. Sandeep [20] (+30 % – one step laser treatment).

4. Conclusion

An experimental study of the influence of various methods of surface treatment of aluminum alloys (chemical, electrochemical, and physical) on the adhesive strength of the metal-polymer interface was conducted.

A combined method of metal surface treatment to increase adhesion to a polymer is proposed, the method includes chemical or electrochemical etching followed by laser treatment. It was shown that complex treatment of the aluminum surface allows to increase the shear strength of composites by 50 %. The laser treatment is effective both before and after chemical or electrochemical treatment. However, a greater effect appears if the laser treatment is carried out after etching. Thus, changing the order between etching and laser treatment allowed to increase the shear strength by 10 %. The effect of laser treatment is most noticeable after etching in $Fe_2(SO_4)_3$. In this case, the increase in shear strength was about 20 %.

Based on the study of changes in the morphology of the aluminum surface during treatment, the true value of the adhesive strength is determined, taking into account the micro- and macro-relief of the surface. It is established that with chemical etching or laser treatment, the value of the true adhesive strength remains virtually unchanged (less than 10 %), however, when using the combined method, its significant increase is observed (about 30 %), and that leads to 50 % increase in shear strength.

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