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# P O L I M E R Y

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## Influence of aluminum alloys surface treatment on the durability of glued joints

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**Abstract:** The aim of the work was to analyze the method of preparing the aluminum surface in terms of the functional properties of glued joints with the use of one-component polyurethane adhesive. Six methods of surface treatment of EN AW-5251 aluminum alloy were tested. In addition, changes in the shear strength of adhesive joints after environmental exposure were determined. The best surface preparation processes were atmospheric plasma and anodizing.

**Keywords:** aluminum and alloys, surface treatment, surface roughness.

### Wpływ obróbki powierzchni stopów aluminium na trwałość klejonych połączeń

**Streszczenie:** Celem pracy była analiza sposobu przygotowania powierzchni aluminium pod kątem właściwości użytkowych połączeń klejonych z zastosowaniem jednoskładnikowego kleju poliuretanowego. Zbadano sześć metod obróbki powierzchni stopu aluminium EN AW-5251. Ponadto określono zmiany wytrzymałości połączeń klejonych na ścinanie po ekspozycji środowiskowej. Najlepszym sposobem przygotowania powierzchni była plazma atmosferyczna i anodowanie.

**Słowa kluczowe:** aluminium i stopy, obróbka powierzchni, chropowatość powierzchni.

The increasing use of aluminum alloys as light materials in various industries requires durable and reliable methods of joining them. The most common methods of joining aluminum are liquid and solid welding, use of mechanical fasteners, gluing, and various combinations of these techniques [1–3]. Glued joints are used, among others, in the automotive industry, but they must meet strict requirements regarding shear strength. In addition, symmetrical consistent destruction is a permitted damage to the glued connector. Damage at the contact of the adhesive with the surface of the substrate and mixed cohesive/adhesive damage is not allowed [4–7].

Adhesive joints are extremely popular due to their low economic costs, but on the other hand, the mechanical strength of these joints is subject to high uncertainty. The disadvantages of adhesive joints are that they are inseparable without damaging the structure, the glued surface should be well prepared, and their properties may change with changes in temperature, humidity, and the environment. Therefore, tests of glued joints are carried out in two directions: mechanical properties and environmental tests [8–13]. The methods of testing adhesive joints described in the literature are used to evaluate the resistance of the adhesive bond and the correctness of its implementation [14–17].

In the case of overlapping glued joints, to obtain good adhesion and mechanical properties, the method of the joined materials surface preparation should be carefully selected [18]. One of the most crucial factors affecting adhesion and cohesion is surface roughness. It can be

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assessed by specifying the roughness profile described, among others, by the parameters  $R_a$  – arithmetic mean deviation of the roughness profile and  $R_q$  – mean square deviation of the roughness profile. The selection of a surface preparation method before making an adhesive joint that increases roughness allows for surface development and obtaining greater intermolecular forces, chemisorption, and diffusion in the adhesive joint. The type of adhesive used (epoxy, methacrylic, polyurethane, etc.), its viscosity and good wettability of the surface are also of immense importance [19–22]. The ability to produce a suitable surface layer is crucial to create a strong adhesive connection. In the case of lap adhesive joints, the highest stress concentration occurs at the edges of the laps. Stress can be lowered by reducing the stiffness of the connection by changing the thickness of the joint or by beveling the side edges of the overlap. As a result, the shear strength of the connection increases [23, 24].

The most used methods of surface preparation for glued joints are mechanical, chemical, electrochemical, and thermal methods. Before making overlapping adhesive joints, the prepared surface and the degree of surface dusting should be assessed [17, 25]. It is also necessary to remove all fatty impurities by degreasing. Organic solvents, alkaline solutions, solutions of surfactants are most often used for this purpose. Electrolytic washers, ultrasonic washers and chamber washers are also finding more and more applications. After the degreasing procedure, depending on the method used, rinsing in water is conducted, followed by thorough drying of the cleaned surface [24, 26–28].

The paper presents the results of adhesive tests of aluminum sheets lap joints subjected to surface treatment various methods. The aim of the research was to determine the effect of surface preparation on the strength of the obtained joints after hardening and after accelerated corrosion tests.

## EXPERIMENTAL PART

### Materials

Aluminum alloy plates EN AW-5251 (composition by weight: 1.7–2.4% Mg, 0.1–0.5% Mn, 0.5% Fe, 0.15% Zn, 0.4% Si, 0.15% Cu, 0.15% Cr and 0.15% Ti) were cut into samples having dimensions of 100×25×2 mm. In accordance with the international standard ISO 4587, a port with a diameter of 4 mm is additionally drilled in each plate. The surface characteristics were conducted, the microstructure of the material was examined, and then glued seams with an overlap of 13 mm were made. Reference samples were made and used for strength and corrosion tests.

Sikaflex®-265 (Sika, Warsaw, Poland) one-component polyurethane adhesive was used to create the adhesive joints. The glue curing mechanism took place by absorbing moisture from the air at a temperature of 10 to 35°C. The adhesive can be used in the temperature range from

–40°C to +90°C. Adhesive strength characteristics: hardness 45ShA, tensile strength 6 MPa (according to CQP 023-1 / ISO 7619-1), tear propagation resistance 12 N/mm (according to CQP 045-1 / ISO34), shear strength 4.5 MPa (according to CQP 046-1 / ISO4587).

### Sample preparation process

Before making the glued joint, the samples of the Al AW-5251 alloy sheet were cleaned of grease contamination using the Teroson FL (Henkel, Düsseldorf, Germany). It is a liquid universal cleaner and solvent based on gasoline, free of fragrances and chlorinated hydrocarbons, characterized by a low content of n-hexane.

Sika® Activator was used to activate the aluminum surface prior to bonding, followed by a solvent-based primer, Sika® Primer-206 G+P.

In order to determine the influence of the aluminum surface preparation on the strength of the glued joint, the aluminum sheets samples were prepared using various surface treatment methods: without surface treatment (sample 1), grinding with an orbital sander with P120 zirconia paper (sample 2), phosphating with Bonderite M-NT 1455-W Conversion Coating Henkel product cloths (sample 3), cleaning in an OpenAir® stream of atmospheric plasma (sample 4), anodizing (sample 5) and chromating (sample 6).

## Methods

### Surface characteristics

The surface profile of the aluminum samples was examined prior to making the adhesive joints. The test was conducted using a MarSurf GD 120 (Mahr, Göttingen, Germany) roughness gauge using the contact method. Five measurements were made for each sample. Based on these tests, the average roughness ( $R_a$ ), the average maximum profile height ( $R_z$ ), the average maximum roughness height ( $R_m$ ), the average roughness height of the core ( $R_k$ ), the average reduced elevation height ( $R_{pk}$ ) and the average reduced elevation depth were determined for all samples ( $R_{vk}$ ). Three-dimensional images of the surface of the coatings were also generated.

### Phosphating the surface

Phosphate coatings were applied using bonderite M-NT 1455-W conversion coating wipes from Henkel. The preparation was rubbed onto degreased metal sheets for about 2 minutes, then left to evaporate for about 10 minutes.

### Plasma jet cleaning

To clean the surface in a plasma stream, atmospheric plasma OpenAir® was used, produced by a Plasmaclean

(Steinhagen, Germany) device equipped with an FG 5001 generator and an FG 5001 plasma head, controlled by a single-axis robot. This device produces a stream of plasma by passing oxygen contained in the air through an electric lye, the temperature of which did not exceed 300°C. The plasma jet exited the nozzle and cleaned the swept surface. The process was carried out at the parameters of air pressure 40 kPa, voltage 290 V, frequency 21 kHz, current 15.9 A. The distance of the nozzle from the surface was 3 mm, the speed of travel was 3.4 m/s with two passes of the nozzle.

### Surface anodizing

Anodized samples were made by applying an oxide coating in an electrolyte containing 20% sulfuric acid, which was sealed in hot water. Anodizing was conducted for 40–70 minutes at a temperature of 15–21°C and a voltage of 12–15 V. The process consisted of the following stages: chemical degreasing, acid etching, brightening, anodizing, sealing in hot water. After each step, the samples were rinsed in distilled water.

### Chromating the surface

Chromating was carried out by immersion in a conventional bath consisting of chromic acid anhydride  $\text{CrO}_3$  in the amount of 250–400 g/L and sulfuric acid (VI)  $\text{H}_2\text{SO}_4$  in the amount of 2.5–4 g/L. The process was carried out in the following stages: chemical degreasing, etching, brightening, chromating, sealing rinsing. After each process, the samples were rinsed in distilled water. Chromating was carried out for 1.5–2 minutes at 18–25°C.

### Characteristics of the adhesive bond

The previously prepared samples of Al AW-5251 aluminum sheet were activated in the first stage. The surface to be bonding was wiped with a cotton cloth soaked in the activating agent and allowed to dry for 30 minutes. Then, with a brush, the primer was applied with one stroke and left to dry for about 40 minutes. The samples prepared in this way were placed in a custom-made

device, specially developed for testing, preparing lap joints of constant dimensions. Using a dispensing gun, a path of polyurethane adhesives was applied, and a second sheet placed on the pad was pressed against the pad (Fig. 1). Bonding was conducted at a temperature of  $20 \pm 2^\circ\text{C}$  and a minimum air humidity of 40% immediately after surface preparation. Curing was carried out for 7 days in a climatic chamber at a temperature of 23°C and 50% air humidity (according to DIN 50014).

### Exposition of samples

After hardening, the samples were exposed to distilled water at 20°C for 7 days. They were then conditioned for 2 hours under curing conditions. The joints prepared in this way were kept at 80°C for 24 hours.

The next aging cycle of glued joints was a 7-days exposure at 70°C in conditions of saturated moisture (Cataplasm). For this purpose, the samples were wrapped in wet cotton cloths and tightly wrapped in aluminum foil. After removal, the samples were conditioned for 2 hours under curing conditions. In the next stage, the joints were cooled down to  $-30^\circ\text{C}$ .

After aging tests according to DVS 1618, the samples were transferred to a salt chamber. Tests of resistance to neutral salt spray (NSS) were carried out in accordance with PN-EN ISO 9227 standards. A Salt-Cab 150 type corrosion test chamber (Biuged Laboratory Instruments Guangzhou Co. Ltd., China) was used for the tests. The capacity of the test chamber of the device was 150 dm<sup>3</sup>, the brine concentration was  $50 \pm 5$  g/L, the pH of the solution was kept within the range of 6.5–7.2, and the test temperature was  $35 \pm 2^\circ\text{C}$ .

The tests were carried out for 360 hours. The weight change of the samples was checked after 3, 5, 8, 10, 13, and 15 cycles, respectively. After accelerated corrosion tests, the bond strength was tested in a testing machine.

### Shear strength

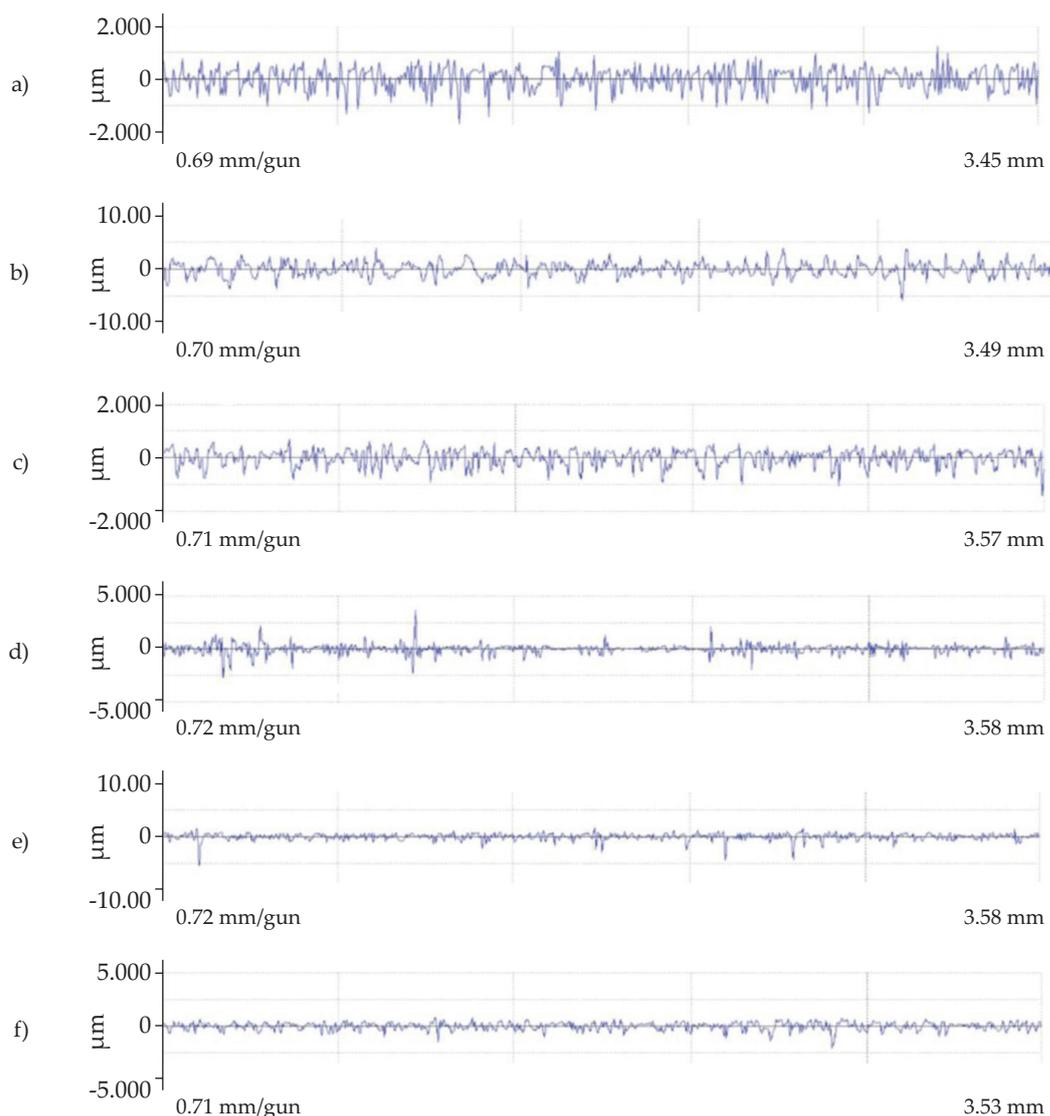
The lap shear strength was performed on a Zwick/Roell Z010 (Ulm, Germany) testing machine. The measurement was carried out in accordance with the PN-EN 1465 standard. Before testing, the samples were conditioned for 2 hours at 20°C and 40% humidity.

## RESULTS AND DISCUSSION

Good adhesion of adhesive joints depends on the roughness of the metal surface. According to the mechanical theory of adhesion, the strength of glued joints increases with increasing material roughness. The authors of some publications indicate that the best strengths are obtained when  $R_a$  ranges from 1.6 to 3.2  $\mu\text{m}$  and  $R_m$  from 7 to 25  $\mu\text{m}$  [19]. Using the surface preparation methods described in the paper, such parameters were not obtained. Only in the case of sample 2 ground with zirconia paper P120,



Fig. 1. Samples after bonding and pressing



**Fig. 2.** Aluminum surface profiles for various surface preparation methods: a) without surface treatment, b) grinding with P120 zirconium paper, c) bonderite phosphating, d) OpenAir® atmospheric plasma, e) anodizing, f) chromating

the  $R_m$  value was 9. The anodized surface had the second largest roughness, deeper valleys were formed in the surface profile  $R_{vk}$ , similarly to sample 2. Sample 4 had a slightly lower roughness. In the case of samples 3 and 6, it was possible to create a surface with similar roughness parameters. Conversion layers, in addition to giving the surface roughness, also provide good anti-corrosion protection.

The results of roughness tests of the EN AW-5251 aluminum alloy sample subjected to various surface preparation methods are presented in Table 1. The surface topography and differences in profiles depending on the surface preparation method are shown in Figure 2. Figure 3 shows a three-dimensional view of the surface of uncoated aluminum samples and after surface treatment.

Based on roughness difference it is possible to compare the bonding strength of the glued joint before and after aging tests.

Samples of aluminum sheet with different roughness profiles, overlapped with polyurethane glue, were subjected to shear strength tests. The aim of these studies was to determine the effect of surface roughness on bonding strength. Figure 4 shows the shear strength of each binder tested with different substrate preparation methods.

The shear strength of the lap joints was 3.7 to 5.7 MPa, depending on the surface preparation. The highest shear strength of lap joints has a sample previously treated in atmospheric plasma, although it has a lower  $R_a$  value than the aluminum surface without surface treatment. The same is true for chromated surfaces. The shear strength of glued joints, previously surface-ground, even though they have the highest  $R_a$  coefficient, is like joints without any treatment. The lowest shear strength is observed with phosphate conversion coatings and anodized surfaces, which is about 4 MPa. In these cases, it is difficult to establish the relationship between surface roughness and

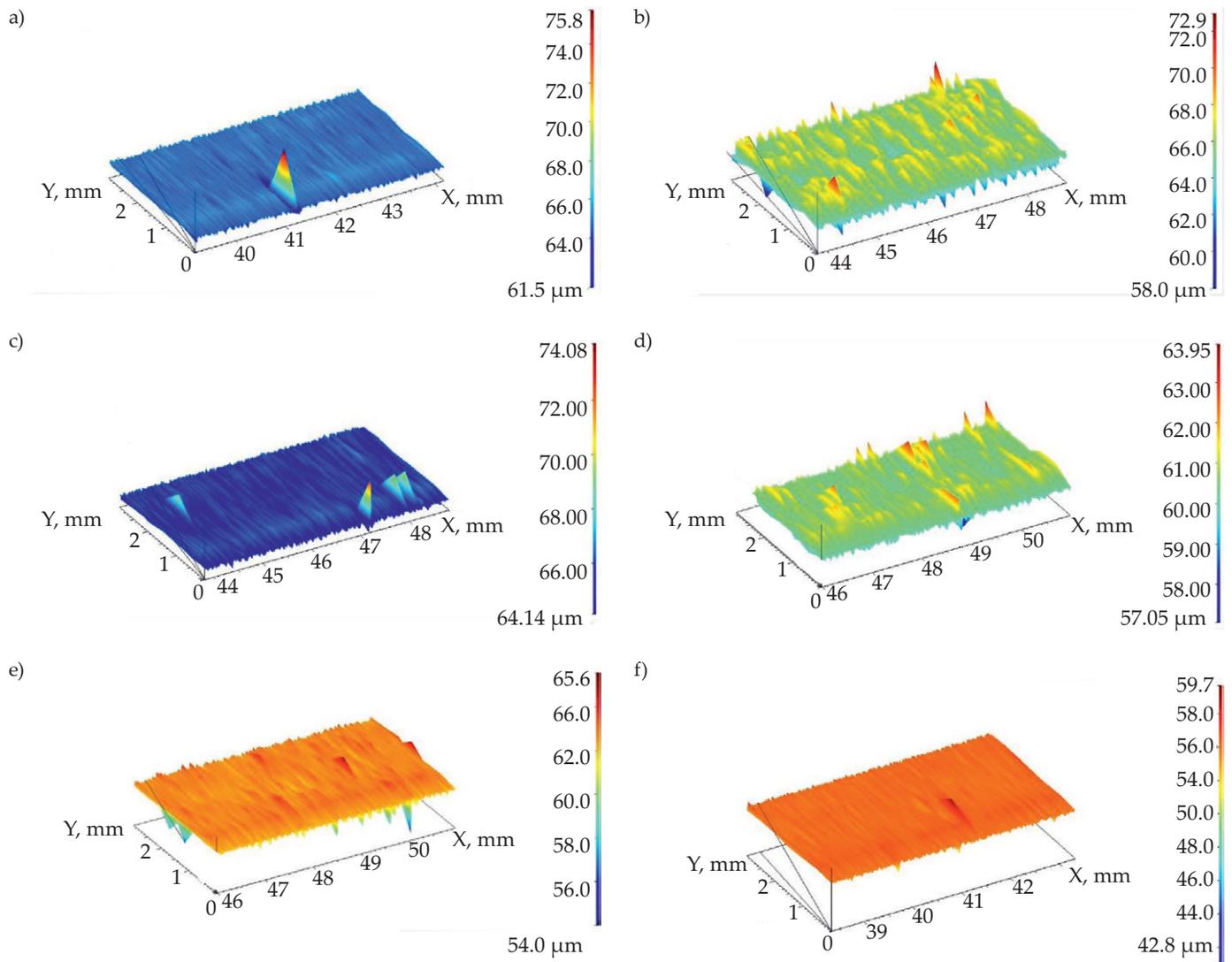


Fig. 3. 3D diagram of aluminum surface: a) without surface treatment, b) grinding with P120 zirconium paper, c) bonderite phosphating, d) OpenAir® atmospheric plasma, e) anodizing, f) chromating

Table 1. Aluminum roughness parameters for various surface preparation methods

Sample	Surface roughness parameters, $\mu\text{m}$					
	$R_a$	$R_z$	$R_m$	$R_k$	$R_{pk}$	$R_{vk}$
1	0.34	2.20	2.49	0.95	0.29	0.52
2	1.03	7.24	9.54	3.10	1.33	1.58
3	0.25	1.61	1.93	0.59	0.17	0.51
4	0.30	3.90	5.89	0.62	0.64	0.79
5	0.44	4.83	6.82	1.32	0.46	1.16
6	0.30	2.00	2.81	0.79	0.18	0.57

tensile strength. The bonding strength is also influenced by the good wettability of the surface with the adhesive. This relationship is described in the literature with various computational models depending on the contact angle of the surface of the materials [25, 26].

In these cases, there is a cohesive or mixed adhesive-cohesive failure with a predominance of cohesive failure. Joint adhesion can be attributed to both chemical bond-

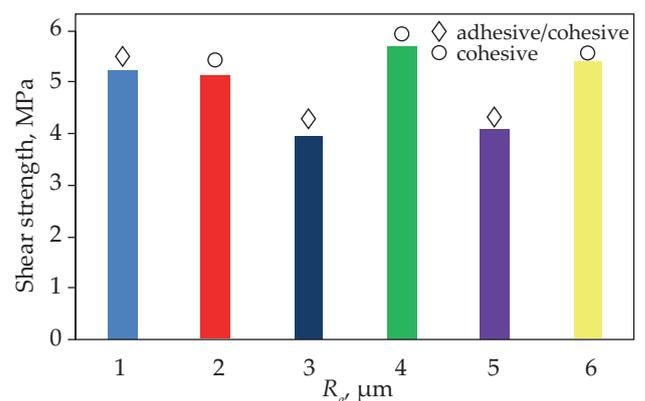


Fig. 4. Dependence of the shear strength on the  $R_a$  value for the tested samples

ing and mechanical bonding during the aluminum alloy joining process [29].

In the case of the tested glued joints, the results of strength tests after exposure to different real environments, i.e., chilly water, 80°C, cataplasm, and neutral salt fog, were also compared (Fig. 5).

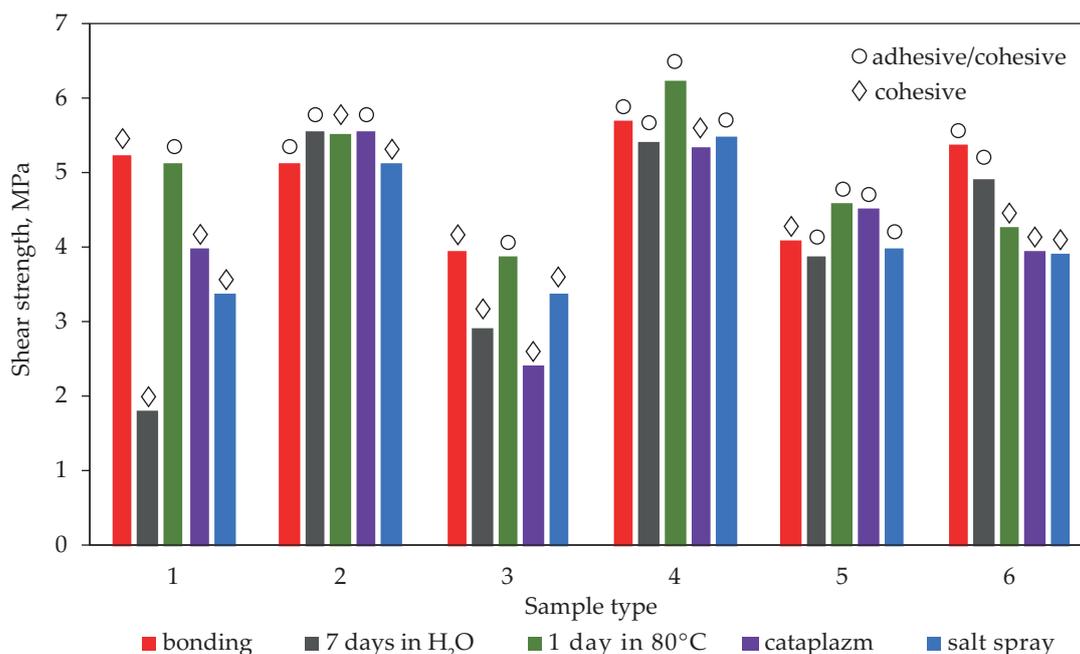


Fig. 5. Shear strength of the tested bonded joints after exposure in different environments

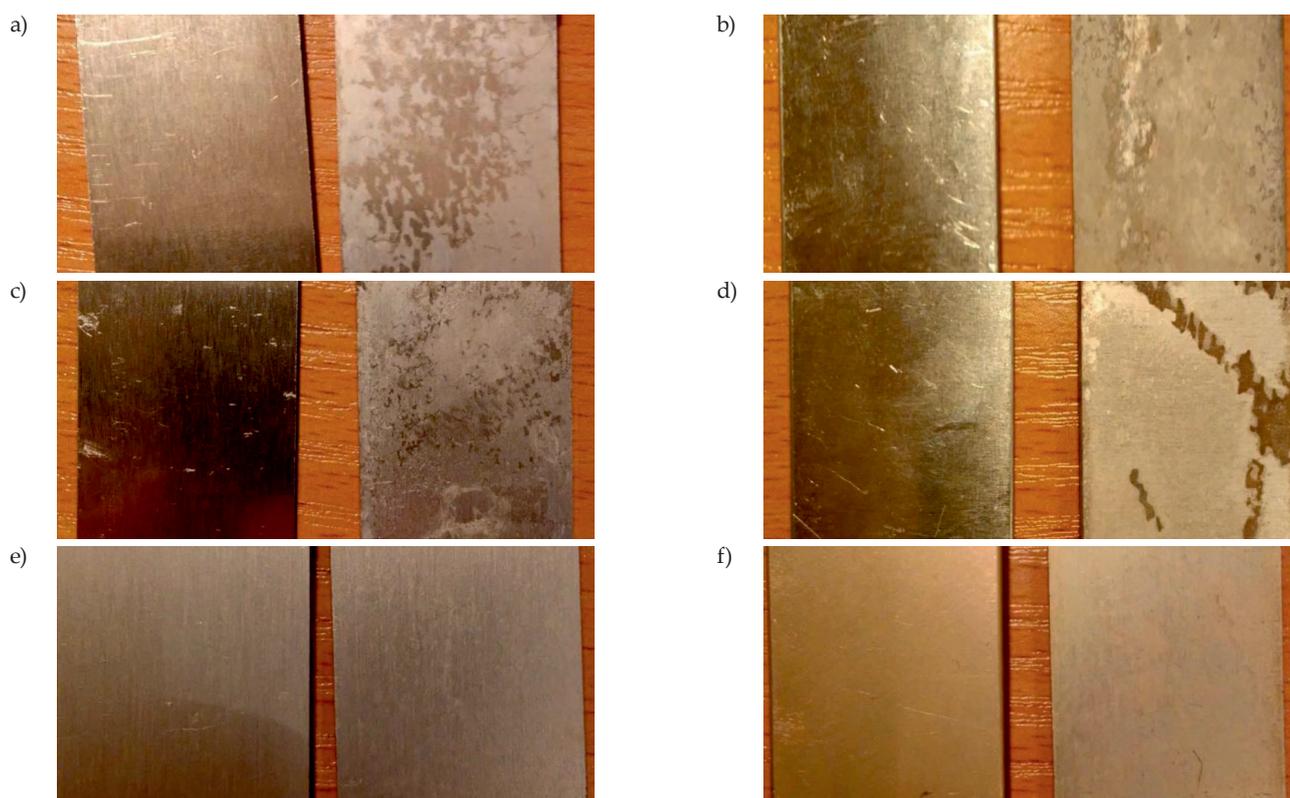


Fig. 6. Surface of aluminum before and after the salt spray test: a) without surface treatment, b) grinding with P120 zirconium paper, c) bonderite phosphating, d) OpenAir<sup>®</sup> atmospheric plasma, e) anodizing, f) chromating

The decrease in shear strength was caused using cataplasm and exposure in a salt spray chamber. The greatest changes in strength were observed in the case of phosphatizing and without prior surface treatment, while the smallest in the case of oxide and chromate coatings. On unprepared, sanded, phosphate and plasma cleaned aluminum joints, corrosion products were clearly visible after exposure to neutral salt spray. The anodic and chrome

coatings became slightly dull after corrosion tests. Photos of the surface of aluminum samples before and after testing in a salt spray chamber are shown in Figure 6.

## CONCLUSIONS

The choice of the type of aluminum surface preparation has a significant impact on the roughness profile of

the bonded material and the adhesion strength of the coating to the substrate, which affects the bond strength between the substrate and the polyurethane adhesive. Shear strength tests have shown an exceptionally low lap strength of 4 to 5.7 MPa for glued joints, and after testing in various environments, they have deteriorated even further. Studies have shown that surface preparation methods by grinding, atmospheric plasma treatment and surface chromating give bonded joints of similar strength. The bond strength slightly increased in the case of plasma by about 11% and chromate by about 5%, although their roughness profiles are lower by about 70% compared to the substrate ground with zirconia paper. After environmental tests, the smallest changes in the shear strength of glued joints are also observed in the same cases. Shear-tested aluminum bonded joints that had been chromated pre-treated showed mixed failure after most environmental tests. Therefore, this aluminum pre-treatment does not seem to be suitable for glued joints. The best ways to prepare the aluminum surface are atmospheric plasma and anodizing. Based on tests carried out on lap joints with Sikaflex®-265 one-component polyurethane adhesive with various methods of aluminum surface preparation, it can be concluded that they have a low shear strength. These types of glued joints can only be used to connect parts that are not subjected to high stresses.

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