The Application of Advanced Surface Preparation Methods in the Adhesive Bonding Process

Abstract: An important part in the process of adhesive bonding is played by the appropriate preparation of surfaces to be subjected to adhesive bonding. The objective of the tests discussed in the article was to identify the effect of various surface preparation methods, including cleaning, grinding, atmospheric plasma treatment and the ATOP method on the strength of adhesive-bonded joints. The tests involved the use of specimens made of aluminium alloy EN AW 5754 as well as specimens made of glass fibre-reinforced epoxy-based plastics. The specimens were subjected to overlap adhesive bonding involving the use of Araldite two-component epoxy adhesive (Huntsman). The article presents results of the static shear test (of the overlap joints) in relation to a given surface preparation method applied before the adhesive bonding process. The highest strength of the adhesive-bonded joints made in the aluminium alloy was obtained in relation to the ATOP method-based treatment. In addition, the above-named method makes it possible to properly prepare a cleaned surface by forming a protective layer ensuring proper surface preparation for several months. In turn, the highest strength of the adhesive-bonded joints made of glass fibre-reinforced plastics was obtained in relation to the surface treatment performed using atmospheric plasma.

Keywords: adhesive bonding, surface preparation methods, surface treatment, adhesive-bonded joints

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Introduction

Presently, adhesive bonding is applied in many industrial sectors including the railway, automotive and aviation industries as well as the production of household appliances and building engineering. Adhesive bonding belongs to the group of special processes and, because of this fact, requires the definition of phenomena affecting adhesion and cohesion. When selecting adhesive appropriate for a given application, it is necessary to pay attention to the mechanism of the bonding and setting (hardening) of adhesives as the mechanisms responsible for proper cohesion and the maintaining of appropriate adhesive bonding conditions, i.e. wettability and adhesion. To ensure the high and repeatable strength of an adhesive-bonded joint it is very important to

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properly prepare surfaces (to be joined) prior to the adhesive bonding process. The objectives of the above-named preparation process are the following [8]:

- improvement of wettability in other words, the spreadability of adhesive (liquid) on the entire surface of the material (solid) to be subjected to adhesive bonding. The obtainment of favourable wettability requires the satisfaction of the following conditions: the pure and degreased surface of materials to be joined, the appropriate proportion of the surface tension of adhesive to that of the element subjected to adhesive bonding, the sufficient mobility of adhesive and the low roughness of the surface,
- improvement of adhesion i.e. the improved ability of adhesive to form a permanent joint with the surface layer of materials subjected to adhesive bonding. In terms of metals, it is important to perform the removal of oxides from the surface of the material (e.g. in relation to steel it is important to remove rust, loosely joined with the material) followed by the direct adhesive bonding of cleaned steel elements. In turn, as regards the adhesive bonding of elements made of aluminium, often covered with a layer of oxides (Al2O3) firmly adhering to the substrate, it is important to remove the above-named layer and allow another layer to form, yet under controllable conditions. In the above-named case, the adhesive is joined with the layer of oxides, firmly adhering to the surface of pure aluminium,
- extension of long-term resistance/strength protection of the material subjected to adhesive bonding against factors responsible for the aging of the joint, and
- ensuring the repeatability of the adhesive bonding process. Pre-adhesive bonding treatment includes two stages, i.e. surface cleaning and preparation.

Cleaning, involving the use of dissolvents or cleaning agents, aims to remove all impurities

and the adsorptive layer from the surface of materials. The cleaning process can be performed manually, e.g. involve the use of special saturated tissues, organic dissolvents, water-based cleaning agents, special substances or may involve immersion, e.g. involve the use of an ultrasonic bath in a special substance, where high frequencies are used to remove impurities from the surface of materials. Cleaning agents should remove all impurities from the surface subjected to adhesive bonding, both water-soluble and fat-soluble ones, and evaporate entirely, not leaving any impurities (sediment) or damaging elements to be joined. For instance, cleaning with acetic acid may lead to corrosion, whereas the cleaning of thermoplastics with alcohol-based substances puts the former at risk of stress scratch formation. The cleaning of surfaces should precede both the mechanical treatment (to prevent the penetration of the surface with impurities during grinding) and chemical treatment (to prevent the contamination of etching agents).

The second stage concerning the preparation of surfaces to be subjected to adhesive bonding involves the use of methods aimed at the uncovering of the surfaces to the level of the base material or its activation. The above-named methods include mechanical, chemical or physical treatment or the application of a primer.

The mechanical treatment is used to remove the surface layer (i.e. an oxide layer in terms of metals and a surface layer usually containing adhesive agents in terms of plastics) to the level of base material. The mechanical treatment usually involves grinding, e.g. with abrasive paper or abrasive blasting with sand or corundum.

Another surface preparation method is the chemical treatment, aimed at the removal of an oxide layer or at the formation of a new layer, e.g. characterised by anticorrosive properties. Apart from providing favourable wettability, the chemical treatment activates surfaces to be joined. Etching involves the application of oxidising acids – nitric acid(V), concentrated

sulphuric acid(VI) and phosphoric acid(V) as well as non-oxidising acids (e.g. diluted hydrochloric acid). In cases of metals, the use of acids not only removes surface (e.g. oxide) layers but also affects the base material. As a result, etching increases roughness and activates metallic surfaces. In addition, the application of oxidising acids results in the oxidation of metallic layers along with the formation of, e.g. phosphate or chromate layers, permanently joined with the substrate. The above-named coatings provide additional protection against corrosion. In view of the fact that etching involves the use of chemically aggressive and noxious substances, after the completion of the process, surfaces subjected to etching should be rinsed with water and dried. Because the effect of etching lasts for a short time, the adhesive bonding process should directly follow surface preparation.

The physical treatment aims to improve wettability and activate the surface. This surface preparation method is used in relation to elements made of plastics, particularly those characterised by low surface energy. One of the above-named methods is the firing of surfaces to be joined with a blue flamed (of propane or acetylene). Within the temperature range of 200°C to 400°C, oxygen atoms are built into the surface, increasing the surface energy of elements, surface wettability and adhesion. However, the treatment effect is short and disappears after several minutes.

Another physical surface preparation method is the plasma-based treatment. Various purposes, e.g. surface activation, cleaning or the application of coatings (also in terms of metals) require various types of plasma (low-pressure plasma, Corona, plasma under atmospheric pressure). Depending on the type of plasma, the surface preparation effect lasts between several hours and several months.

Industry usually utilises the low-pressure plasma treatment performed in a combustion chamber. The ignition of plasma is triggered by high voltage or microwave in vacuum. Low-pressure plasma is rated as cold plasma and can be used while preparing elements of complex shapes. Because of the fact that the process takes place in a closed chamber, it is repeatable and fully controlled. During the low-pressure plasma treatment, plasma particles penetrate deeply the surface of an element, thus providing it with a long-lasting effect. The method is limited by the dimensions of the vacuum chamber, which, in turn, restricts the size of a workable element.

In the Corona treatment, rated among hot plasma-based methods, plasma is generated as a result of high voltage discharges in atmosphere between two electrodes. The above-named type of plasma is useless for the treatment of conductors and elements having complicated shapes. Advantages of the Corona treatment include ease and low cost. The effect of Corona treatment lasts for a very short time. As a result, the adhesive bonding process should directly follow surface preparation.

The plasma treatment under atmospheric pressure involves the high voltage-triggered ignition of plasma in a nozzle. The stream of gas transports plasma from the nozzle onto the surface of an element to be joined. The fact that plasma is generated in the nozzle enables its control. An advantage of the method is its usability when preparing elements on production lines. The plastic surface activation effect lasts several weeks. The atmospheric plasma-based



Fig. 1. Treatment with plasma under atmospheric pressure [8]

treatment (Fig. 1) can also be applied to clean metals, yet in such situations, the adhesive bonding process should directly follow surface preparation.

Another method used for surface activation before adhesive bonding is the application of an additional substance, i.e. a primer. The primer - type of adhesive (necessary application of is used to improve the wettability and adhesability of materials and, additionally, to partial- - condition of the surface of elements subjectly even the roughness of surfaces to be joined, acting as a "connector" between the surface of - requirements concerning an element subjectan element and the layer of adhesive. The use of primers improves the adhesion of adhesives on surfaces characterised by low surface energy. e.g. poorly adhesible plastics, e.g. polypropylene and polyethylene.

A new surface preparation method is the so-called Alternative Surface Cleaning Method (ASCT). The method is used for the cleaning of surfaces of various materials, particularly metal elements, including aluminium alloys, copper, bronze, magnesium, steel, cast iron etc. The surface cleaning process includes the removal of organic impurities, e.g. grease, and inorganic impurities, such as oxides, hydroxides and salts, usually simultaneously present on the surfaces of elements subjected to adhesive bonding. The cleaning process is accompanied by the formation of a layer

protecting the prepared surface against various agents and factors. The abovenamed protective layer makes it possible to perform adhesive bonding up to several months following the performance of the surface preparation process. The surface treatment process can involve the immersion of elements in the aqueous solution or by spraying the entire surface with the cleaning substance. Time needed for the cleaning of the surface and the formation of the protective layer depends on surface impurity and material and lasts several minutes. In contrast with the chemical method, the alternative surface cleaning technology does not require

the application of aggressive chemicals; the cleaned and activated surface is not at risk of getting damaged.

The selection of the surface treatment method should depend on the following factors:

- _ type of material subjected to adhesive bonding,
- a primer, if any),
- ed to adhesive bonding (e.g. rusty surface),
- ed to adhesive bonding (e.g. safety class).

Tests performed at Łukasiewicz Research Network - Instytut Spawalnictwa revealed that the application of an appropriate surface preparation method before the performance of the adhesive bonding process significantly affects the strength and quality of subsequently obtained adhesive-bonded joints. The effect of individual surface preparation methods on the strength of adhesive-bonded joints in relation to the specimens made of aluminium alloy EN AW 5754 and subjected to adhesive bonding with epoxy adhesive Hysol 9466 is presented in Figure 2 [9]. The process of etching led to the obtainment of the highest strength of adhesive-bonded joints (amounting to 35 MPa), by twice higher than the strength of the joints cleaned with isopropyl alcohol.

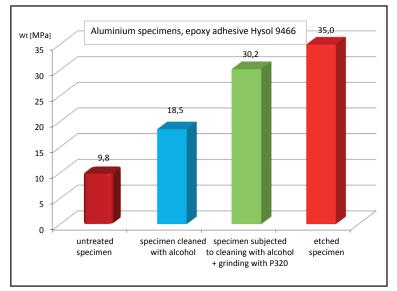


Fig. 2 Effect of the aluminium alloy surface preparation on the strength of the joints subjected to adhesive bonding involving the use of epoxy adhesive Hysol 9466 [9]

In the tests presented in the article, pre-adhesive bonding surface preparation involved the use a new method (instead of etching), i.e. the alternative surface cleaning technology (ASCT), where the substance used for the cleaning of the surface and, at the same time, providing protection through a special layer, is safe both for environment and human health.

Testing methodology

The tests aimed to identify the effect of various surface preparation methods, including cleaning (with alcohol), grinding (with abrasive paper), plasma (AD) and the ASCT-based method on the strength of adhesive-bonded joints. Presented below are the aforementioned methods.

- 1. Cleaning of specimens specimens were cleaned using tissues saturated with isopropyl alcohol; the specimens were subjected to adhesive bonding directly after surface preparation.
- 2. Mechanical treatment specimens were cleaned with isopropyl alcohol and, afterwards, subjected to (crosswise) grinding with abrasive paper having a granularity of P120 followed by repeated cleaning with isopropyl alcohol; the specimens were subjected to adhesive bonding directly after surface preparation.
- 3. ASCT-based method specimens were immersed for several minutes in the aqueous solution of substance TAB1003PA1 heated up to a temperature of 60°. Afterwards, the specimens were rinsed with distilled water and dried with air heated to a temperature of 200°C; the specimens were subjected to adhesive bonding directly after surface preparation. Some of the specimens (additionally described in the article) were stored at room temperature and protected against contamination for 8 months following surface preparation and then subjected to adhesive bonding.
- 4.

atmospheric pressure - specimens were cleaned with isopropyl alcohol and, subsequently, subjected to treatment with plasma under atmospheric pressure; the specimens were subjected to adhesive bonding directly after surface preparation.

The shear strength tests of the adhesive-bonded joints involved the specimens made of aluminium alloy EN AW 5754 and the epoxy-based glass-reinforced plastic (GRP) specimens. The tests specimens, having the dimensions of 25 mm \times 100 mm, were made in accordance with the PN-EN 1465:2009 standard. The thickness of the specimens made of aluminium alloy amounted to 2.0 mm, whereas that of the GRP specimens amounted to 1.5 mm. The specimens were subjected to overlap adhesive bonding, where the overlap had the dimensions of 12.5 mm × 25.0 mm (Fig. 3).

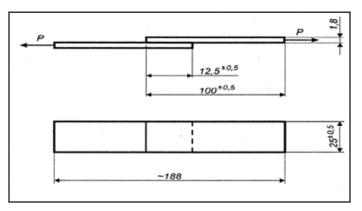


Fig. 3 Schematic diagram of the overlap adhesive-bonded joints used in the shear strength tests [13]

The static shear tensile tests involving the overlap adhesive-bonded joints were performed using an INSTRON 4210 testing machine and a travel rate of 5 mm/min.

The adhesive-bonded joints were made using epoxy adhesives characterised by high resistance to shear strength (and chemical), i.e. Araldite 2011 and Araldite 2013 (Huntsman). The above-named adhesives are intended for the adhesive bonding of metals, glass, rubber and rigid plastics. Table 1 presents open times of the adhesives and selected values of shear strength in relation to the aluminium alloy and the GRP, Treatment with AD plasma under in accordance with the adhesive data sheet.

	Open time (100 g at 20°C)	Shear strength
Araldite 2011	50 min	– 26 MPa for aluminium – 11.5 MPa for GRP
Araldite 2013	100 min	– 18 MPa for aluminium – 8.5 MPa for GRP

Table 1 Specifications of the adhesives used in the tests

The scope of the tests included configurations presented in Table 2. Five overlap joints were made in relation to each of the combinations. The test results presented in the tables and diagrams are mean values based on five tests. The strength tests of the adhesive-bonded joints were performed one week after the performance of adhesive bonding.

Table 2 Scope of	the tests [12]
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Material sub- jected to adhe- sive bonding	Surface preparation method	Adhesive	
EN AW 5754	Cleaning with isopropyl alcohol		
	Cleaning and grinding with abrasive paper P120	Araldite	
+ EN AW 5754	ASCT-based treatment (5 min)	2011	
	AD plasma-based treatment		
	Cleaning with isopropyl alcohol		
EN AW 5754	Cleaning and grinding with abrasive paper P120	Araldite	
+ EN AW 5754	ASCT-based treatment (5 min)	2013	
	AD plasma-based treatment		
	Cleaning with isopropyl alcohol		
GRP	Cleaning and grinding with abrasive paper P120	Araldite	
	ASCT-based treatment (5 min)	2011	
	AD plasma-based treatment		
GRP + EN AW 5754	GRP – cleaning with isopropyl alcohol Al – cleaning with isopropyl alcohol		
	GRP – ASCT-based treatment (1 min) Al – ASCT-based treatment (1 min)	Araldite	
	GRP - cleaning with isopropyl alcohol Al – ASCT-based treatment (1 min)	2011	
	GRP – AD plasma-based treatment Al – AD plasma-based treatment		

The tests were preceded by measurements of the surface energy of the materials subjected to adhesive bonding. The contact angle was measured using a BTG LABS SA3001 tester (Fig. 4), whereas the measurement of surface energy involved the application of test inks.



Fig. 4 Contact angle measurement device [15]

The surface energy measurements involving the aluminium specimens revealed that, in relation to the untreated (uncleaned) specimens, the contact angle amounted to 80°, whereas the surface energy was 30 mN/m. After degreasing with isopropyl alcohol the contact angle amounted to 60°, whereas the surface energy was 50 mN/m. The highest value of surface energy (measured directly after the treatment) was obtained in relation to the specimen subjected to the AD plasma-based treatment, i.e. 72 mN/m; the contact angle being 16°. In turn, surface energy measured several minutes after the treatment started to decrease and amounted to 68 mN/m, whereas the contact angle increased up to 28°.

The surface energy measured on the specimen made of the GRP, the surface of which was cleaned with isopropyl alcohol, amounted to 44 mN/m. After the AD plasma-based treatment, surface energy increased to 70 mN/m. In relation to the plastics, the effect resulting from the AD plasma-based treatment lasted several weeks following the surface treatment.

Test results

Tests involving the specimens made of aluminium alloy EN AW 5754

Results of the static shear test involving the adhesive-bonded joints on the specimens made of aluminium alloy EN AW 5754 and subjected to adhesive bonding directly after preparation are presented in Table 3 and Figures 5–6.

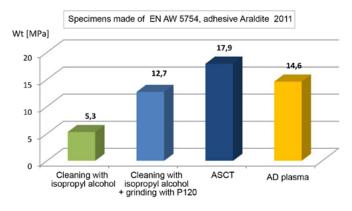


Fig. 5 Shear strength of the adhesive-bonded joints in relation to pre-adhesive bonding surface preparation; Araldite 2011 [12]

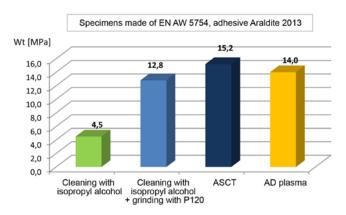


Fig. 6 Shear strength of the adhesive-bonded joints in relation to pre-adhesive bonding surface preparation; Araldite 2013 [12]

The shear strength test results concerning the specimens made of aluminium revealed that, in relation to both epoxy adhesives (Araldite 2011 and Araldite 2013), the highest strength was obtained following the ASCT-based treatment. In comparison with the specimens cleaned with alcohol and subjected to grinding, the strength of the specimens subjected to the ASCT-based treatment increased by 40% in relation to Araldite 2011 and by 19% in relation to Araldite 2013. Apart from high strength, the use of the

Table 3. Shear strength of the adhesive-bonded joints on the specimens made of aluminium alloy EN AW 5754 in relation to the surface preparation method [12]

Adhesive	Surface treatment	Shear strength [MPa]	Standard deviation
Araldite 2011	Cleaning with isopro- pyl alcohol	5.3	0.8
	Cleaning and grind- ing with abrasive paper P120	12.7	1.2
	ASCT-based treat- ment (5 min)	17.9	0.6
	AD plasma-based treatment	14.6	3.7
Araldite 2013	Cleaning with isopro- pyl alcohol	4.5	0.7
	Cleaning and grind- ing with abrasive paper P120	12.8	1.4
	ASCT-based treat- ment (5 min)	15.2	0.3
	AD plasma-based treatment	14.0	4.6

ASCT-based method also led to the obtainment of high repeatability. In relation to the Araldite 2011 adhesive, the standard deviation amounted to 0.6, whereas in Araldite 2013, the standard deviation amounted to 0.3.

High strength values were also obtained in relation to the AD plasma-based cleaning method, where the surface of the specimens made of aluminium alloy was both cleaned and activated. In the above-named case, the standard deviation was the highest in comparison with other surface treatment methods and amounted to 4.6 in relation to Araldite 2011 and 3.7 in relation to Araldite 2013.

The aluminium surface preparation through cleaning and grinding (i.e. the most common aluminium treatment method) enabled the obtainment of strength amounting to 12.7 MPa (in relation to Araldite 2011) and 12.8 MPa (in relation to Araldite 2013). In turn, the use of the AD plasma-based method increased strength by 15% (14.6 MPa) – in relation to Araldite 2011 and by 8% (14.0 MPa) – in relation to Araldite 2013.

Tests involving the specimens made of glass-reinforced plastics (GRP)

Results of the static shear test involving the adhesive-bonded joints on the specimens made of glass-reinforced plastic (GRP) and subjected to adhesive bonding directly after preparation are presented in Table 4 and Figure 7.

Table 4. Shear strength of the adhesive-bonded joints on the specimens made of glass-reinforced plastics (GRP) in relation to the surface preparation method (GRP) [12]

Adhesive	Surface treatment	Shear strength [MPa]	Standard deviation
Araldite 2011	Cleaning with iso- propyl alcohol	15.6	0.5
	Cleaning and grinding with abra- sive paper P120	13.6	2.6
	ASCT-based treat- ment	18.0	0.8
	AD plasma-based treatment	19.1	0.7

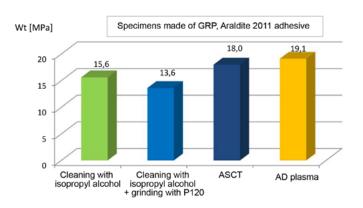


Fig. 7. Effect of pre-adhesive surface preparation method on the strength of the joins made of the GRP specimens; Araldite 2011 [12]

In relation to the adhesive bonding of the GRP specimens subjected to adhesive bonding with Araldite 2011, the highest strength was obtained in relation to the AD plasma-based treatment, i.e. 19.1 MPa. The above-named value was higher by 40% than that of the specimens prepared using isopropyl alcohol and grinding with abrasive paper P120. High strength was also obtained in relation to the joint subjected to the ASCT, i.e. 18.0 MPa. The above-named value was by 32% higher than that of the

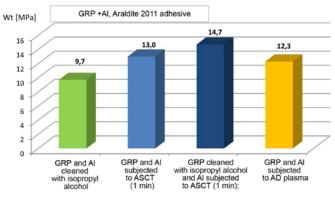
specimens subjected to cleaning and grinding with abrasive paper. In terms of glass-reinforced plastics, the application of grinding could adversely affect the strength of joints.

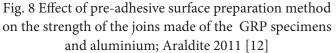
Tests involving specimens made of GRP joined with aluminium alloy EN AW 5754

Results of the static shear test involving the adhesive-bonded joints on the specimens made of glass-reinforced plastics (GRP) and subjected to adhesive bonding (using Araldite 2011) with the specimens made of aluminium alloy EN AW 5754 directly after surface preparation are presented in Table 5 and Figure 8.

Table 5 Shear strength of the adhesive-bonded joints made of glass-reinforced plastics (GRP) and aluminium alloy EN AW 5754 in relation to the surface preparation method [12]

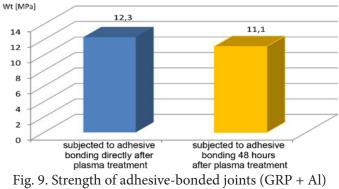
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Adhesive	Surface treatment	Shear strength [MPa]	Standard deviation
Araldite 2011	cleaned GRP cleaned Al	9.7	1.6
	GRP subjected to ASCT (1 min) Al subjected to ASCT (1 min)	13.0	2.2
	cleaned GRP Al subjected to ASCT (1 min)	14.7	0.2
	GRP subjected to AD plasma Al subjected to AD plasma	12.3	0.3





As regards the adhesive bonding of the GRP specimens with the aluminium alloy, the highest strength was obtained in relation to the

joints, where the specimens made of GRP were cleaned with isopropyl alcohol and the specimens made of aluminium were subjected to the ASCT-based treatment. In the above-named case, strength increased by 51% in comparison with that of the specimens only cleaned with alcohol. A high value of 13.0 MPa was obtained where both the specimens made of GRP and those made of aluminium were subjected to the ASCT-based treatment. In turn, the AD plasma-based treatment, where the specimens were subjected to adhesive bonding directly after surface preparation, resulted in a strength increase of 26% in comparison with the strength of the specimens only cleaned with alcohol. The specimens subjected to adhesive bonding 48 hours after plasma treatment revealed a decrease in strength from 12.3 MPa to 11.1 MPa (Fig. 9). The test results demonstrated that the obtainment of joints characterised by high strength required the performance of the adhesive bonding process directly after treatment.



prepared using the AD plasma method in relation to time following treatment [12]

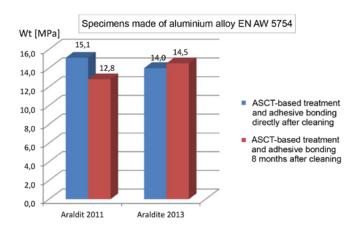
Tests involving specimens made of ASCTprocessed aluminium alloy EN AW 5754 and subjected to adhesive bonding 8 months after preparation

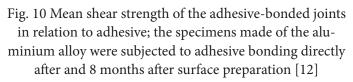
According to the producer, the ASCT method used in the tests not only cleans metal surfaces, providing the high quality of adhesive bonded joints but also provides prepared surfaces with a protective layer lasting for several months. Table 6 and Figure 10 present test

results concerning the specimens made of aluminium alloy EN AW 5754, prepared using the ASCT method (the specimens were cleaned for 10 minutes) and subjected to adhesive bonding directly after preparation and 8 months after preparation.

Table 6. Specimens made of the aluminium alloyand cleaned in the ASCT substance [12]

Adhesive	Shear strength [MPa]	
Adhesive bonding directly after surface preparation		
Araldite 2011	15.2	
Araldite 2013	14.0	
Adhesive bonding 8 months after surface		
preparation		
Araldite 2011	12.8	
Araldite 2013	14.5	





In terms of the specimens subjected to the ASCT-based treatment, subjected to adhesive bonding 8 months after surface preparation, the protective layer provided the cleaned surface with appropriate protection; the mean strength of the joint fell by 15%, amounting to 12.8 MPa for the Araldite 2011 adhesive, and was comparable with that of the specimens subjected to mechanical cleaning and grinding (12.7 MPa). In relation to Araldite 2013, the strength of the specimens subjected to adhesive bonding 8 months after surface preparation was comparable with that of the specimens subjected to adhesive bonding directly after the surface preparation treatment.

Conclusions

The tests justified the formulation of the following conclusions:

- The highest strength of adhesive-bonded joints was observed in relation to the specimens made of aluminium alloy EN AW 5754 subjected to the ASCT. The above-named strength was higher by 40% (in relation to the Araldite 2011 adhesive) and by 19% (in relation to Araldite 2013) than the strength of the joints prepared by cleaning and grinding with abrasive paper P120.
- 2. In terms of the adhesive-bonded joints made of glass (fibre)-reinforced plastics (GRP), the highest shear strength was obtained after plasma treatment performed under atmospheric pressure. The above-named shear strength was higher by 40% than that of the joints prepared by cleaning and grinding with abrasive paper P120.
- 3. As regards the joining of glass-reinforced plastics (GRP) with aluminium alloy EN AW 5754, high strength was obtained where the aluminium alloy specimens were subjected to the ASCT and the specimens made of plastics were cleaned using alcohol. In the above-named case, the strength of the joints increased by 51% in comparison with the strength of the specimens only treated with alcohol.
- 4. The use of plasma-based treatment under atmospheric pressure in relation to elements made of aluminium alloy requires the adhesive bonding process to be performed directly after the surface treatment process, whereas the application of the ASCT-based method provides surfaces to be subjected to adhesive bonding with a protective layer lasting for several months.

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