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IMPACT OF ADHESIVE TYPE AND ABRASIVE WEAR USED IN THE SURFACE PREPARATION PROCESS ON THE STRENGTH OF STEEL SHEET ADHESIVE JOINTS

WPŁYW RODZAJU KLEJU I ZUŻYCIA ŚCIERNIWA STOSOWANEGO W PROCESIE PRZYGOTOWANIA POWIERZCHNI NA WYTRZYMAŁOŚĆ POŁĄCZEŃ KLEJOWYCH BLACH STALOWYCH

Abstract

The aim of the paper was to present issues related to the determination of the influence of selected technological factors: the method of surface preparation and type of adhesive on the strength of adhesive joints made of steel sheet C45. As a method of surface preparation the process of shot-blasting with the use of three types of abrasives differentiated in terms of the degree of its wear was used. The adhesive bonds were made with the use of two two-component epoxy adhesive compositions based on Epidian 57 epoxy resin and PAC and Z-1 curing agents combined with the resin in the recommended proportions. The measurements of roughness and topography of surfaces prepared for the bonding process were also carried out. After the curing process, strength tests of adhesive bonds were performed on the Zwick/Roell 150 testing machine, according to PN-EN 1465 standard. It was noted, among others, that with increasing wear of the abrasive used in the surface preparation process, the value of adhesive bonds strength decreased and the higher strength of adhesive bonds was characterized by adhesive joints made with Epidian57/Z-1/100:10 adhesive. The obtained results were subjected to statistical analysis.

Keywords: adhesive joints, C45 steel sheet, tensile strength, shot blasting

Streszczenie

Celem artykułu było przedstawienie zagadnień związanych z określeniem wpływu wybranych czynników technologicznych: sposobu przygotowania powierzchni oraz rodzaju kleju na wytrzymałości połączeń klejowych wykonanych z blachy stalowej C45. Jako sposób przygotowania powierzchni wykorzystano proces śrutowania z wykorzystaniem trzech rodzajów ścierniw zróżnicowanych pod względem stopnia jego zużycia. Połączenia klejowe wykonano przy użyciu dwóch dwuskładnikowych kompozycji klejowych epoksydowych bazujących na żywicy epoksydowej Epidian 57 oraz utwardzaczy PAC i Z-1 łączonych z żywicą w zalecanych proporcjach. Przeprowadzono także pomiary chropowatości oraz topografii powierzchni przygotowanych do procesu klejenia. Po procesie utwardzania dokonano prób wytrzymałościowych połączeń klejowych na maszynie wytrzymałościowej Zwick/Roell 150, zgodnie z normą PN-EN 1465. Zauważono m.in., że wraz ze wzrostem stopnia zużycia ścierniwa użytego w procesie przygotowania powierzchni, wartość wytrzymałości połączeń klejowych malała, a wyższą wytrzymałością charakteryzowały się połączenia klejowe wykonane przy użyciu kleju Epidian57/Z-1/100:10. Uzyskane wyniki poddano analizie statystycznej.

Keywords: połączenia klejowe, blacha stalowa, wytrzymałość, śrutowanie

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1. Introduction

There are many methods of joining structural components. For this purpose, assembly methods such as welding, riveting, welding, soldering or adhesive bonding can be used. It is important to choose the right method to ensure that the joints have the expected properties. However, the selected method must not adversely affect the specific properties of the structure, which include, but are not limited to: the dimension of the construction, shape, functionality, usability, reliability, aesthetics, modularity and universality [1–4].

One of the oldest and most frequently used methods of joining construction materials is adhesive bonding. Adhesives, as adhesive plastics, have the ability to create adhesive forces on the components to be bonded. The process of adhesive bonding consists in applying a thin layer of adhesive substance between the surfaces to be bonded, which connects them by means of adhesive forces and cohesion force, i.e. the force of internal coherence of the materials [5]. In addition, adhesives used in joints provide corrosion protection and can be used as sealants. A number of factors influence the effectiveness of the bonding process and the properties of the joints. Technological, construction, material and operational factors are among them. These factors include: the method of surface preparation of the materials to be bonded, the type of adhesive together with the method of its application on the surfaces to be bonded, as well as the curing conditions of the adhesive joint depending on the type of adhesive (temperature, time and pressure) and the conditions of seasoning [6, 7]. Changes to these factors during the bonding process may affect the properties of certain joints in different ways. However, due to the specification of the joints under consideration, it is necessary to conduct studies to analyse the effects of these factors on specific cases and applications. A change in these factors for a particular joint may affect the properties of the joint, e.g. another material, in a slightly different way, including its strength properties [8, 9].

Analyzing the technological process of adhesive bonding, special attention should be paid to the preparation of the surface. The process of surface preparation consists in removing the surface layer of oxides and other impurities occurring in the form of lubricants, dusts, sediments, oils, microorganisms, moisture, etc [3]. The main distinguishing features are mechanical and chemical cleaning, as well as special treatments dedicated to specific materials. Mechanical cleaning is, for example, machining with an abrasive bulk tool, sandblasting, shotblasting, flame burning or machining [10–12]. Chemical cleaning is carried out using solvents such as: petrol, acetone, benzene, various types of detergents, trichloroethylene [13–15]. The surface preparation process significantly affects the adhesive properties of the bonded materials and the strength of the adhesive joints. Thanks to appropriate surface preparation, it is possible to obtain properly made adhesive joints and to ensure adequate joint strength, of course, while also complying with other structural and technological conditions [8]. Appropriate surface preparation increases the durability of the joint and determines the reliability of the joint.

In this paper the analysis of the influence of abrasive wear used in the surface preparation process and the effects of the adhesive type on selected strength aspects of C45 steel sheets adhesive joints is considered.

2. Studies methods

2.1. Characteristics of adhesive joints and bonded material

In the experimental studies, single-overlapping adhesive joints were made. The joints were made of low carbon steel sheets C45 (1.0503). C45 steel is one of the higher quality structural steels. Unalloyed quality steel is used for heat-treatment, easy to process but hardly weldable. It is mainly used in machine elements and equipment of medium load e.g. tools, knives, shafts, screws, discs, levers, gears, crankshafts, spindles, woodworking tools [12, 16–18]. The chemical composition and some physical properties of the used material (according to PN EN 10020:2003) are presented in Table 1.

Table 1. Chemical composition and physical properties of C45 steel [19]

Stainless steel C45			
Chemical composition, %		**Physical properties**	
C	0.44	Rm	638 MPa
Mn	0.55		
Si	0.21		
P	0.01	Re	369 MPa
S	0.02		
Cr	0.16		
Ni	0.24	Hardness	255 HB
Cu	0.07		

The subject of the study were single overlap adhesive joints loaded on shear as shown in Figure 1.

The length of the overlap was assumed to be 15 mm, and the thickness of the adhesive layer to be about 0.2 mm. The area of adhesive and the thickness of the adhesive joint were verified before the strength tests.

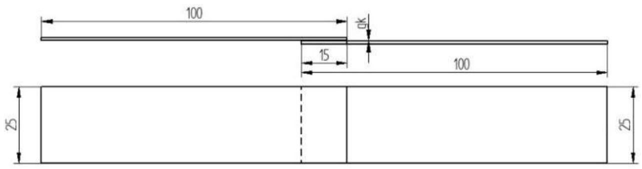

Fig. 1. Single overlap adhesive bond of steel sheet C45

2.2. Method of steel sheet surface preparation

Surface preparation is one of the first stages in the process of making permanent adhesive bonds. Several processes connected with cleaning and geometric development of joined elements' surfaces in the presented studies. The surface development of the used samples was achieved by mechanical shotblasting process, where the used abrasive was corundum of various degrees of wear. An abrasive is a fine grain of an abrasive material, which is the basis for the construction of abrasive tools. Electrocorundum (Al_2O_3) is one of the most common and cheapest abrasives. It is a synthetic version of a mineral called corundum. Corundum in its pure form is used only for polishing, sometimes for tool sharpening [20, 21]. Corundum grains have sharp edges and controlled properties. It consists mainly of aluminium oxide. The shotblasting process was carried out with an AUER shotblasting machine. The blasting pressure was 0.6 MPa and the nozzle distance was 100 mm. During the shotblasting process, the samples were divided into 3 groups because 3 types of abrasive were used, differentiated in the level of wear. The abrasive wear was estimated as small, medium and high, depending on the time it was working. The classification into particular variants of shotblasting is presented in Table 2.

Table 2. Shotblasting variants according to wear of the abrasive used to prepare steel sheet surface

Shotblasting	Level of wear	Operating time of abrasive
Variant 1	Small	24 hours (1 days)
Variant 2	Medium	496 hours (3 months)
Variant 3	High	992 hours (6 months)

The next step in the surface preparation process, after machining, was degreasing the surface. Degreasing is one of the most frequently used surface preparation operations performed just before the joining process, because it removes all kinds of dust and grease impurities, often remaining after previous processing [14, 19]. Degreasing of steel sheet surfaces intended for bonding was carried out with technical acetone by spraying three times. For the first two replications the degreasing agent was removed with a dust-free fabric, while after the third spraying the samples were allowed to self-evaporate and completely dry.

2.3. Characteristics of the used adhesive

Two types of epoxy adhesive compositions were used to make adhesive joints, which were the subject of the study. The base for both adhesives was Epidian 57 epoxy resin. This resin occurs in the form of viscous liquid of light yellow colour and characteristic smell of styrene. Its basic features are: very good mechanical strength, high resistance to chemical substances such as oils, greases, acids, etc., proper adhesion to the substrate and good hardness of the obtained adhesive coating, very good adhesive joints of various materials such as ceramics, metal, glass, wood, possibility of hardening at ambient temperature. The epoxy number of Epidian 57 is 0.40 mol/100g, density at 20°C is in the range of 1.14-1.17 g/cm3, and viscosity at 25°C - 13 000 - 19 000 mPas [22, 23].

The resin must be properly mixed with the cure agent in order to cross-link and harden. The first adhesive composition used for the adhesive bonds consisted of the resin and PAC hardener. PAC hardener is a viscous liquid with an amber colour and characteristic amine smell. When used with epoxy resins, the PAC hardener is used for joining elements exposed to deformation, e.g. bonding thin sheets, joining rubber with metal, in compositions for flooding elements in electrical and electronic engineering [19, 24]. The preparation of the composition was based on mixing Epidian 57 epoxy resin and PAC hardener at a weight ratio of 100:80. In the further part of the paper the composition determination – Epidian57/PAC/100:80 was used. The second composition was prepared with the use of Z-1 hardener. This is a viscous liquid of light yellow colour and characteristic smell for triethylenetetraamine. The Z-1 hardener is used primarily in connection with low-molecular weight epoxy resins and for joints exposed to deformation, such as joining thin sheets, joining rubber with metal or plywood [19, 24]. It is also used to harden liquid epoxy resins. The composition was prepared at a proportion by weight to resin ratio of 100:10 - Epidian57/Z-1/100:10.

Epidian57/PAC/100:80 adhesive composition is much more slow-bonded than Epidian57/Z-1/100:10 composition. PAC curing agent is one of the slow-reacting hardeners. After about 12 hours the initial hardening of the composition takes place and after 72 hours it reaches the hardening of 80 - 90%. The total cure lasts from 7 to 14 days. This process can be accelerated by increasing the temperature after the first curing step. In case of gelation of compositions containing Z-1 hardener this time is about 35 minutes at ambient temperature. Initial cure (degree of cure 80-90%) is achieved after 48 hours. However, the total cure lasts 7-14 days. It should also be noted that adhesive compositions containing PAC hardener are characterized by higher elasticity, higher impact resistance and lower resistance to elevated temperature than compositions hardened with Z-1 curing agent.

The preparation of two-component adhesive compositions was based on a precise mixing of epoxy resin with a selected hardener at an appropriate weight ratio. The OP-2 laboratory scale with the accuracy of 0.01 g was used to weigh the necessary amount of adhesive compositions components. Both compositions were mixed with a mechanical turbine mixer with two blades during 3 minutes at a speed of 460 rpm. The adhesive compositions were prepared just before the process of adhesive bonding. They were applied manually to one of the joined surfaces with a serrated polymer float, keeping constant thickness over the whole joined surface.

2.4. Conditions for performing and testing adhesive bonds

The bonding process was carried out in laboratory conditions at 26 ± 1°C with a humidity of 32%. The samples were conditioned to harden the joint for 7 days under 0.14 MPa pressure. After this time, the samples were subjected to strength tests. A tensile shear test was performed on a Zwick/Roell Z150 testing machine. The test was carried out in accordance with DIN EN 1465 standard at a crosshead speed of 5 mm/min. For each variant of surface preparation and for each type of adhesive, 10 single overlap adhesive bonds were made. Additionally, prior to the bonding process, the surface roughness and topography were measured using the T8000 RC 120-140 from Hommel--Etamic. Measurements were taken for 3 randomly selected samples from each variant of surface preparation.

3. Results of research

3.1. Roughness measurements results

The measurements of roughness and surface topography were performed in accordance with PN-EN ISO 13565-2:1999. The length of the measurement distance was ln = 4.8 mm and the elementary distance lr = 0.8 mm. The analyzed surface roughness parameters were: Ra - arithmetic mean of the ordinates of the roughness profile, Rt - total height of the roughness profile and Rp - height of the highest elevation of the profile [25, 26].

Figure 2 shows the influence of the abrasive wear level used in the surface preparation process on the Ra roughness parameter. Considering the results obtained, it can be observed that the value of the arithmetic mean of the ordinate decreases with increasing wear of the abrasive used for surface preparation of the samples. The use of an abrasive that has operated for 6 months (variant 3) resulted in a 44% decrease in the Ra parameter in relation to the Ra parameter value for variant 1, where the abrasive wear rate was small.

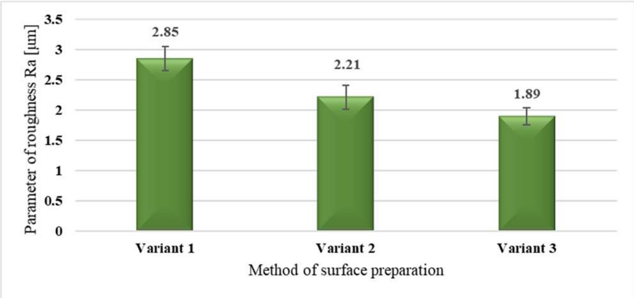

Fig. 2. Effect of abrasive wear level of the abrasive used in the surface preparation process on the arithmetic average of the ordinates roughness profiles (Ra)

Considering the height parameters of the surface roughness profile Rt, Rp, it should be noted that similarly to the Ra parameter, the value of the parameters decreases with increasing abrasive wear, as shown in Figure 3.

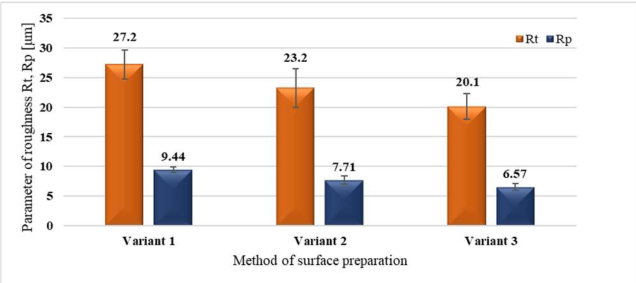

Fig. 3. Effect of the abrasive wear level of the abrasive used in the surface preparation process on the height parameters of the roughness profile (Rt, Rp)

Shotblasting affects the lack of direction of the surface geometric structure (Fig. 4). The shotblasting operation has resulted in many peaks and cavities with sharp peaks and valleys on the surface, which can be better detected in the surface profilograms (Fig. 5).

Taking into account the presented results of roughness measurements and surface topography, it can be expected that such a shaped surface may be a surface with good adhesion properties. This can be proved by the undirected geometric structure of the surface characterized by numerous peaks and valleys, which positively influences the penetration and anchoring of the adhesive [19, 27, 28].

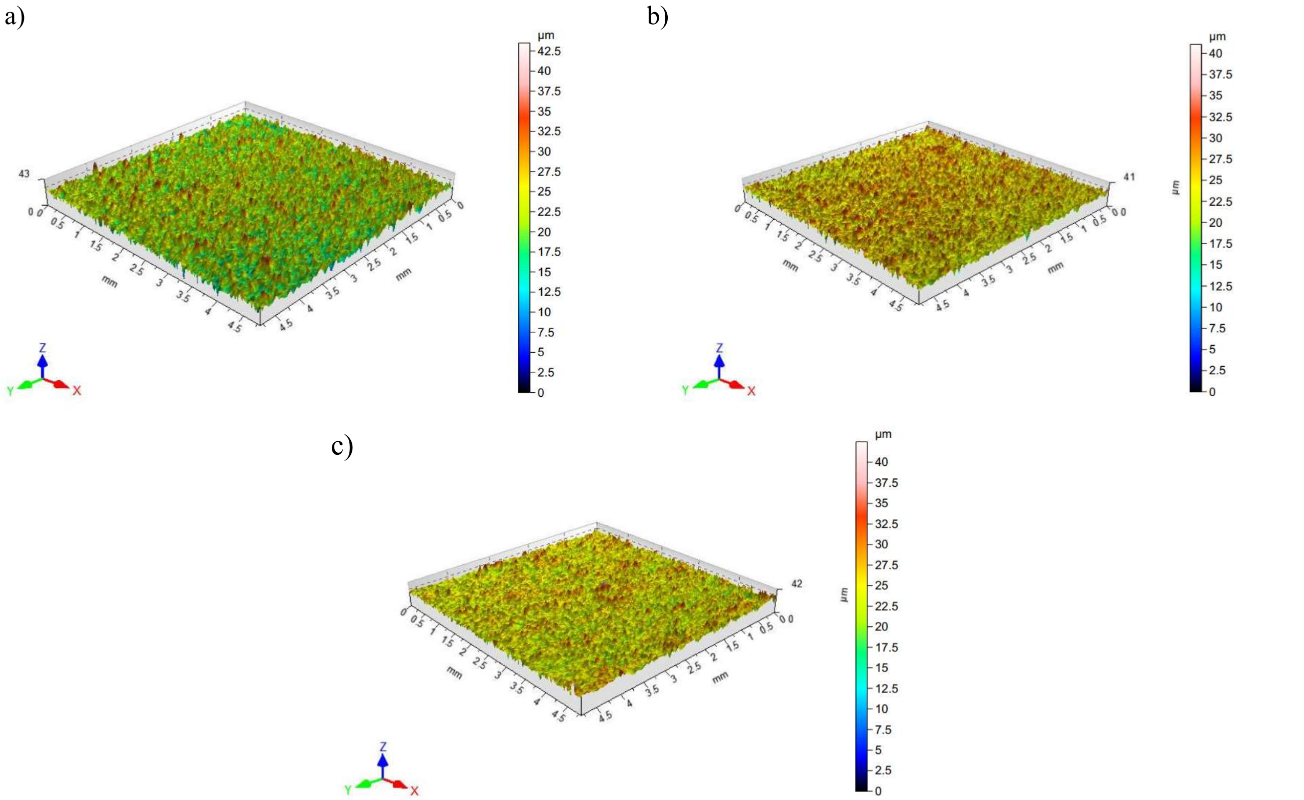

Fig. 4. Influence of the abrasive wear level of the surface preparation process on the surface topography (a) variant 1 - small abrasive wear level, (b) variant 2 - medium abrasive wear level, (c) variant 3 - high abrasive wear level

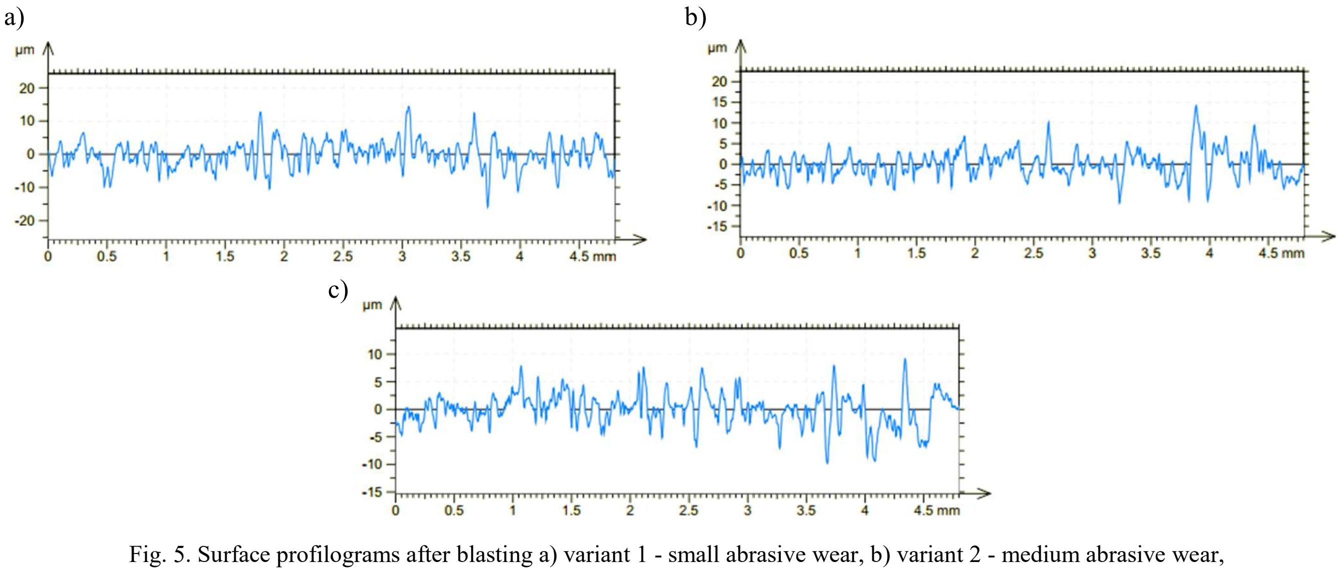

Fig. 5. Surface profilograms after blasting a) variant 1 - small abrasive wear, b) variant 2 - medium abrasive wear, c) variant 3 - high abrasive wear

3.2. Strength test results

After a period of time, when the adhesive joint achieved complete curing, the adhesive joints were subjected to destructive strength tests. The results are shown in Figure 6.

On the basis of the strength results obtained, it can be observed that with the increase in abrasive wear level, the strength of adhesive bonds decreased. For both adhesives, the highest strength was obtained in the case of samples whose surfaces were subjected to 1 shotblasting variant, i.e. with the lowest abrasive wear. The adhesive bonds made with Epidian57/ Z-1/100:10 adhesive were characterized by higher strength. This may result from different properties in comparison with the second adhesive used - Epidian57/PAC/100:80, which is characterized by

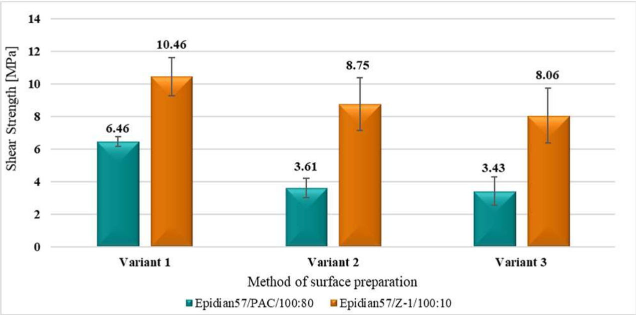

Fig. 6. Results of shear strength of steel sheet adhesive joints made with two epoxy adhesives due to the abrasive wear level of the abrasive used in the surface preparation process

higher elasticity but also higher viscosity. For this reason, the penetration of the adhesive into the surface cavities along the length of the adhesive overlap may have been more problematic. It should be noted, however, that despite the differences between the strength of joints made with both adhesives, the distribution of these values is similar in both cases - the value of strength decreases with increasing abrasive wear. An important aspect worth emphasizing is also the repeatability of the results. In the case of samples prepared for the bonding process with variant 1, the repeatability of the results is much higher than in the case of the other two variants. The value of standard deviation for Epidian57/PAC/100:80 adhesive is 0.30 MPa for variant 1 (which constitutes 4.71% of average strength), 0.61 MPa for variant 2 (which constitutes 17.02% of average strength), 0.87 MPa for variant 3 (which constitutes 25.5% of average strength). For Epidian57/Z-1/100:10 the values of standard deviations were as follows: for variant 1 - 1.16 MPa (which constitutes 11.18% of mean strength), for variant 2 - 1.62 MPa (which constitutes 18.53% of mean strength), for variant 3 - 1.67 MPa (which constitutes 20.80% of mean strength). It can be noticed that higher repeatability of the results was obtained in the case of joints made with Epidian57/PAC/100:80 adhesive.

3.3. Statistical analysis of the obtained results

The strength of adhesive bonds is an important factor in the evaluation of such bonds. However, in order to be able to compare the results obtained, it is necessary to analyze them statistically.

At the beginning, the distribution normality of the obtained results was checked with the assumed confidence level of $\alpha = 0.05$. For this purpose, the Shapiro-Wilk test was used. Results of this test are presented in Table 3.

The analysis of the conformity of empirical distribution with the normal distribution by Shapiro--Wilka test did not reject the hypothesis of the normality of strength distributions of the tested adhesive bonds for the analysed methods of surface preparation ($p>\alpha$). The next step was to check the variances homogeneity using the Levene test, which also did not reject the hypothesis about the equality of variances. Therefore, ANOVA statistics were carried out in order to verify the occurrence of significant differences in the influence of abrasive wear on the strength of adhesive joints in the process to prepare the surfaces of the joined elements and the type of adhesive. Post-hoc Tukey test was carried out to determine homogeneous groups of distribution. The results of this test are presented in Table 4.

Table 3. Normality test - adhesive joints' strength

Shotblasting	Type of adhesive	Shapiro--Wilk statistics W	Probability level p	Normality of distribution
Variant 1	Epidian57/ PAC/100:80	0.836298	0.184825	Yes
	Epidian57/ Z-1/100:10	0.908799	0.476064	Yes
Variant 2	Epidian57/ PAC/100:80	0.786330	0.079880	Yes
	Epidian57/ Z-1/100:10	0.788513	0.083166	Yes
Variant 3	Epidian57/ PAC/100:80	0.841345	0.199353	Yes
	Epidian57/ Z-1/100:10	0.843504	0.205812	Yes

Table 4. The post-hoc Tukey's test designating homogenous groups

Shotblasting	Type of adhesive	Average shear strength [MPa]	Homogenous groups	
			a	b
Variant 1	Epidian57/ PAC/100:80	6.46	****	
	Epidian57/ Z-1/100:10	10.46		****
Variant 2	Epidian57/ PAC/100:80	3.61	****	
	Epidian57/ Z-1/100:10	8.75		****
Variant 3	Epidian57/ PAC/100:80	3.43	****	
	Epidian57/ Z-1/100:10	8.06		****

On the basis of the obtained results it can be noticed that the strength results for the samples bonded with Epidian57/PAC/100:80 adhesive for all shotblasting variants are in one group and for Epidian57/Z-1/100:10 adhesive in the other group. This means that at a given confidence level $\alpha = 0.05$ the abrasive wear level does not significantly affect the change of strength properties of the obtained joints. In this case, the type of adhesive has a bigger influence.

4. Conclusions

The conducted research concerned the influence of abrasive wear in the abrasive processing process on the static strength of overlapping adhesive joints of C45 steel sheets. Variable factors in the study were: the method of surface preparation for adhesive bonding and the type of adhesive. As a method of surface preparation, the shotblasting process was used with the use of three types of abrasives differentiated in their wear level. Two types of two-component epoxy adhesive compositions based on Epidian 57 epoxy resin with PAC and Z-1 curing agents added to the resin in appropriate proportions, according to the manufacturer's recommendations, were used as a binder. Measurements of roughness and topography of surfaces prepared for the bonding process were also carried out. Based on the tests carried out, it can be seen that:

- the roughness parameters decrease with increasing wear of the abrasive used in the surface preparation of the samples to be bonded,
- shotblasting is a process that positively influences the development of the surface to be bonded, which can be a surface with good adhesive properties,
- with the increase in the level of abrasive wear used in the process of surface preparation, the value of strength of adhesive bonds decreased,
- higher strength was characterized by adhesive bonds made with the use of a less lightweight adhesive, i.e. Epidian57/Z-1/100:10. However, higher repeatability of results was obtained in the case of bonds made with Epidian57/PAC/100:80 adhesive.

To summarize, it should be stated that mechanical processing has a significant impact on the strength of adhesive joints made of structural sheet. This is due to the formation of more micro roughness, which contributes to a better anchoring of the adhesive in such a surface, bearing in mind that this is also related to the type of adhesive used. The degree of abrasive wear and tear in the process of shotblasting the connected elements resulted in a deterioration of the obtained adhesive strength results of steel sheet joints. However, the statistical analysis showed that at the set confidence level $\alpha = 0.05$ these differences are not considered significant. In this case, the type of adhesive has more influence.

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