THE EFFECT OF THE SURFACE PREPARATION METHOD ON THE ULTIMATE STRENGTH OF A SINGLE LAP ADHESIVE JOINTS OF SELECTED CONSTRUCTION MATERIALS

Wpływ sposobu przygotowania powierzchni na wytrzymałość doraźną zakładkowych połączeń klejowych wybranych materiałów konstrukcyjnych

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A b s t r a c t: The aim of the article was to present issues related to the determination of the influence of the surface preparation method on the strength of adhesive joints made of three types of construction materials: structural steel C45, aluminium alloy EN AW-1050A and stainless steel 1.4401. The surfaces of the analysed materials were prepared by machining with three different abrasive tools of different gradations: P120, P220, P400. Adhesive joints were prepared using the E53/Z-1/100:10 epoxy adhesive composition. After the process of adhesive joint curing, destructive tests were carried out on the Zwick/Roell Z150 strength machine, in accordance with PN-EN 1465 standard, which determined the shear strength of the analyzed joints. During the tests it was observed that the most advantageous method of surface preparation is treatment using P220 grit abrasive.
K e y w o r d s: structural steel C45, aluminium alloy EN AW-1050A, stainless steel 1.4401, shear strength, adhesive joints

S t r e s z c z e n i e: Celem artykułu było zaprezentowanie zagadnień związanych z określeniem wpływu sposobu przygotowania powierzchni na wytrzymałość połączeń klejowych wykonanych z trzech rodzajów materiałów konstrukcyjnych: stali konstrukcyjnej C45, stopu aluminium EN AW-1050A oraz stali nierdzewnej 1.4401. Powierzchnie analizowanych materiałów zostały przygotowane poprzez obróbkę trzema różnymi narzędziami ściernymi różnej gradacji: P120, P220, P400. Połączenia klejowe przygotowano z użyciem kompozycji klejowej epoksydowej E53/Z-1/100:10. Po procesie utwardzania spoiny klejowej przeprowadzono badania niszczące na maszynie wytrzymałościowej Zwick/Roell Z150, zgodnie z normą PN-EN 1465, dzięki którym wyznaczono wytrzymałość na ścinanie analizowanych połączeń. W trakcie badań zaobserwowano, że najkorzystniejszym sposobem przygotowania powierzchni jest obróbka wykorzystująca ścierniwo ziarnistości P220.
S ł o w a k l u c z o w e: stal konstrukcyjna C45, stop aluminium EN AW-1050A, stal nierdzewna 1.4401, wytrzymałość na ścinanie, połączenia klejowe

Introduction

The design of structural adhesive joints sometimes poses difficulties due to the fact that the short-term strength of such bonds depends on many factors: material, construction, technological and operational [8, 13, 24]. Among these factors, one of the most important is how to prepare the surfaces of the materials to be joined. The proper preparation of the surface in the process of bonding determines the correct execution of the adhesive joint and obtaining the appropriate strength of the joint, and, consequently, determines the correct operation of the joint in specific conditions, as well as increases the resistance of the joint to various operating factors [1, 2, 32, 39, 39]. This stage determines the correct operation of the joint to a large extent. It should ensure the strongest possible adhesive bonds in the adhesive joint. For this purpose, it is necessary to [7, 14]:

- remove all impurities from the surfaces of the elements to be bonded (such as: grease, dust, grease, microorganisms, gas bubbles, loosely bonded corrosive layers), which can significantly reduce the adhesive bond strength,
- get the appropriate surface "roll-out",
- achieve good activation of the surfaces of the elements being joined.

The choice of surface preparation method depends on many factors, including the type, properties and stereometric structure of the surface of the materials being joined [4, 33]. Depending on the properties of the materials, technical and technological conditions, workshop possibilities and others, the surface preparation process can consist of different operations:

- cleaning and degreasing the surface,
- special processing,
- actions immediately prior to establishing the joint.

Degreasing is designed to remove contaminants from the surface which include: oil, grease, moisture and other undesirable substances that make it difficult for further processes to activate the surface. Surface degreasing can be performed manually in case of unit production or in case of complicated shape of the degreased element. This type of degreasing is relatively imperfect and time- and labour-intensive, therefore degreasing is most often carried out in baths of solvents or their vapours. Various solvents (e.g. acetone, gasoline) can be used for degreasing. When using water for degreasing, it is necessary to check whether the material being treated absorbs water - whether it is hydrophilic. If so, the pre-treatment must be carried out by removing the water, e.g. by drying, which can be done in the ambient air, in an air stream (can be heated to 40-50°C), in an inert gas atmosphere (e.g. nitrogen, argon) or in a chamber dryer.

The purpose of special surface treatment is to develop the surface properly and increase its physical and chemical activity. Special surface treatments can include mechanical, chemical, laser, plasma, electrochemical and other methods [11, 12, 16, 35, 36].

Mechanical methods include: abrasive machining, abrasive blasting (e.g. sandblasting, shot blasting), peening, scraping, brushing, grinding [27]. These methods enable the surface's geometric structure to be constituted, but do not guarantee good surface activation [31].

Chemical methods allow for appropriate development of the surface and surface layer with a chemical composition that ensures high surface physicochemical activity in relation to the adhesive used. Chemical treatment usually consists in pickling the surface of elements to be glued in baths of appropriate composition and temperature. The etching time is also important.

The application of special primers is recommended for some materials in the final stage of surface preparation. These agents contain chemically active functional groups that react both with the adhesive and with the surfaces to be bonded. This operation has a positive effect on increasing the adhesive strength of the adhesive joint [31].

A properly prepared surface for the bonding process should be characterized:

- no impurities reducing adhesion,
- good wettability of the adhesive,
- the ability to produce interphase bonds,
- stability for the assumed conditions and the life of the connection,
- the repeatability of the obtained properties,
- the presence of activators/properdisposition agents (if required).

The change of factors influencing the quality of the bonding process may affect the properties of certain joints in different ways. The issues of influence of these factors on the strength of adhesive joints are described in many works [5, 37]. However, due to the specification of the joints under consideration, it is necessary to conduct research related to the analysis of the influence of these factors in relation to specific cases and applications. The change of these factors for a particular joint may affect the properties of the joint, e.g. another material, including its strength properties, in a slightly different way.

The choice of the method of preparing the surface for the bonding process depends on many factors, but one of the most important is the type of material analysed. With regard to low-alloy steel, there are recommendations for the application of surface preparation method presented in some works [25, 28]. Due to the properties of C45 steel for ease of machining, it is recommended to grinding, abrasive machining, sandblasting, shot blasting, superfinishing and polishing.

In terms of surface preparation of aluminium alloys, chemical and electrochemical treatments are recommended [3, 10, 15, 23]. Often used treatment is anodizing, chromating and phosphating. The first operation in the process of surface preparation of aluminium and aluminium alloys is degreasing, which can be carried out using various techniques and degreasing agents [26, 30].

Equally often the recommended treatment for aluminium alloys is mechanical. This is carried out using abrasive bulk tools. It results in a geometrical development of the surface, which significantly increases mechanical adhesion, which is related to the increase in the active contact surface of the adhesive with the bonded material. When using mechanical processing, the key aspect is the appropriate selection of the abrasive grain size. Too small a grain may cause the impurities to wash away on the surface, while coarse a grain creates too deep scratches, which may cause changes in the properties of the surface layer.

A significant impact of mechanical processing on the strength of adhesive bonds can be observed in the results of research published in the previous studies [6, 29, 33]. On the basis of the results presented in the paper [29], it can be seen that in the case of aluminium alloy, better results in relation to the strength of joints were obtained using electrochemical treatment. However, when choosing the surface treatment, the dimensions and shape of the structure must be taken into account in addition to the properties of the material from which the joint is made. This is due to factors such as costs and complexity of the preparation process. Mechanical treatment will be relatively cheaper and less invasive and less harmful as a method of surface preparation than electrochemical treatment. It should be stressed that e.g. inappropriate composition of the pickling bath as well as too long pickling time contribute to the high porosity of the conversion layer, which may result in a deterioration of the corrosion resistance.

With regard to stainless steel, the surface can be subjected to both chemical and mechanical treatment [18]. One of the chemical methods is etching, whose technological parameters can be adjusted to the properties of the alloys.

The aim of the research was to compare the ultimate strength of single-lap adhesive joints, made using three types of construction materials: structural steel C45, aluminium alloy EN AW-1050A and stainless steel 1.4401, whose surfaces for the bonding process have been prepared by mechanical treatment with abrasive grit tools of different gradations.

Methodology of experimental testing

Adhesive joints being the subject of the tests were made of metal sheets with dimensions: 100x25x2 mm. Single-lap joints were made. The thickness of the adhesive layer was 0.1 ± 0.02 mm. The scheme and geometry of the joints are shown in Figure 1.

To make the connections, sheets of three types of construction materials were used, which are often used in machine building [9, 34]. Structural steel sheet C45, aluminium alloy EN AW-1050A and stainless steel 1.4401 were used. The mechanical and physical properties of the materials used are presented in Table 1.

The surface of the samples to be bonded has been prepared with the use of mechanical abrasive treatment with gritting and degreasing tools. This treatment consisted in roughening the surface with graded abrasive paper: P120, P220 and P400. 30 rotary movements with the abrasive papers of the aforementioned grit sizes were performer on the surface of each samples. After mechanical working the samples were degreased by rubbing-through two times with use of a extraction gasoline. The drying time after degreasing was 2 minutes.

For the adhesive joints, the Epidian 53 epoxy resin adhesive composition and Z-1 curing agent in the amounts of 100 grams of resin and 10 grams of hardener were used (composition designation E53/Z-1/100:10).

Epidian 53 (producer: CIECH S.A.) is a mixture of the epoxide resin made of bisphenol A and epichlorohydrin (Epidian 5) and styrene. It is characterized by low adhesiveness (at 25 °C: 900–1500 mPa·s) and lower density than Epidian 5 (at 20°C: 1.11–1.15 g/cm^3). Epidian 53 is characterized by high strength at a temperature of about 110°C [17]. Its modifications are used in joining glass laminates. Due to great electro-insulation and resistance properties, it can be used in radio engineering, aviation, and optics.

Curing agent Z-1 (producer: CIECH S.A.) is an aliphatic amine. It has a viscosity of 20-30 mPa·s (at 25 °C) and a density of 0.978-0.983 g/cm^3 (at 20 °C). It is mainly used in compositions with low-molecular-weight epoxy resins and preparations based on them. It is used in industrial, specialized and consumer applications.

Weighing of components of adhesive compositions was performed with the use of KERN CKE 3600-2 electronic laboratory balance with measurement accuracy of 0.01 g. The composition was mixed mechanically using a paddle mixer, with the speed of 460 rpm in 2 minutes. The adhesive was prepared directly before the joining process. The adhesive was applied manually in a thin layer on two joined surfaces using a polymer spatula.

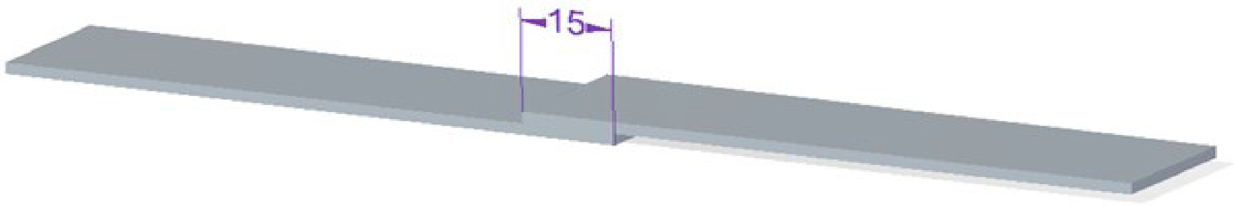

Fig. 1. Single-lap adhesive joint to be tested

Table 1. Mechanical and physical properties of steels used in tests [28, 18, 20, 21, 22]

The properties	Structural steel C45	Aluminium alloy EN AW-1050A	Stainless steel 1.4401
Tensile strength Rm	560-850 MPa	65-95 MPa	500-700 MPa
Yield strength, Re	275-490 MPa	20 MPa	≥195 MPa
Elongation, A	14-17 %	26 %	>40 %
Shear moduls, E	198-207 GPa	69 GPa	200 GPa
Thermal capacity, cp	482 J·kg^{-1}·K^{-1}	899 J·kg^{-1}·K^{-1}	500 J·kg^{-1}·K^{-1}
Thermal conductivity, λ	49.4 W·m^{-1}·K^{-1}	229 W·m^{-1}·K^{-1}	15 W·m^{-1}·K^{-1}
Hardness	≤219 HB	20 HB	<230 HB

The joining process was performed at a temperature of 25±2°C with a humidity of 27±2%. Under the same conditions, a one-stage curing process of the adhesive joint was carried out, using a pressure of 0.20 MPa. There were 10 adhesive joints prepared for each type of material and for each method of surface preparation. In total, 90 adhesive bonds were prepared for strength testing.

After a curing time of 7 days, the adhesive joints were subjected to strength tests on the Zwick/Roell Z150 strength machine in accordance with PN-EN 1465:2009 [19] at a test speed of 5 mm/min. Shear strength results of the tested adhesive joints were obtained during the tests.

Results and analysis of the obtained research results

The average value and standard deviation were calculated for each batch of samples. During the analysis of the experimental results, the extreme values for a specific batch of samples were rejected. Extreme results (too high and too low in relation to other values) were rejected when the differences between the results were large. Differing results could have resulted, among other things, from defects in the weld structure that could have occurred during its execution.

The average results of shear strength of adhesive joints of C45 structural steel sheets, EN AW-1050A aluminium alloy and 1.4401 stainless steel, the surfaces of which were machined with three gradations of P120, P220 and P400, are shown in Figure 2.

Analyzing the obtained results of the shear strength test of single-lap adhesive joints of C45, EN AW-1050A and 1.4401 stainless steel, the surfaces of which were prepared using P120 grade sandpaper, it can be seen that the highest strength was obtained in the case of joints made of C45 structural steel (4.06 MPa). Lower by about 38% strength was obtained in the case of the other two materials - the strength of adhesive joints of aluminum alloy EN AW-1050A was 2.50 MPa, and stainless steel 1.4401 - 2.53 MPa.

In the case of adhesive joints of the analyzed materials, the surface of which was prepared with the use of P220 gradation sandpaper, the highest strength was characterized by joints made of C45 (5.10 MPa) structural steel sheets. The lowest shear strength was obtained in the case of joints made of 1.4401 (2.73 MPa) stainless steel sheets. In the case of adhesive joints made with aluminum alloy sheets

EN AW-1050A, shear strength of 3.05 MPa was obtained.

Comparing the obtained results of shear strength of adhesive joints made of materials whose surfaces were prepared with the use of P400 graded abrasive paper, it can be seen that the highest strength was obtained in the case of C45 - 4.14 MPa stainless steel joints. Stainless steel connections 1.4401 - 1.66 MPa had the lowest strength.

The highest repeatability of results was obtained in the case of joints, which were also characterized by the highest strength among the analysed materials, i.e. made of C45 structural steel sheets.

However, in order to be able to accurately assess the differences between the shear strength results obtained in individual groups, it was necessary to carry out a more

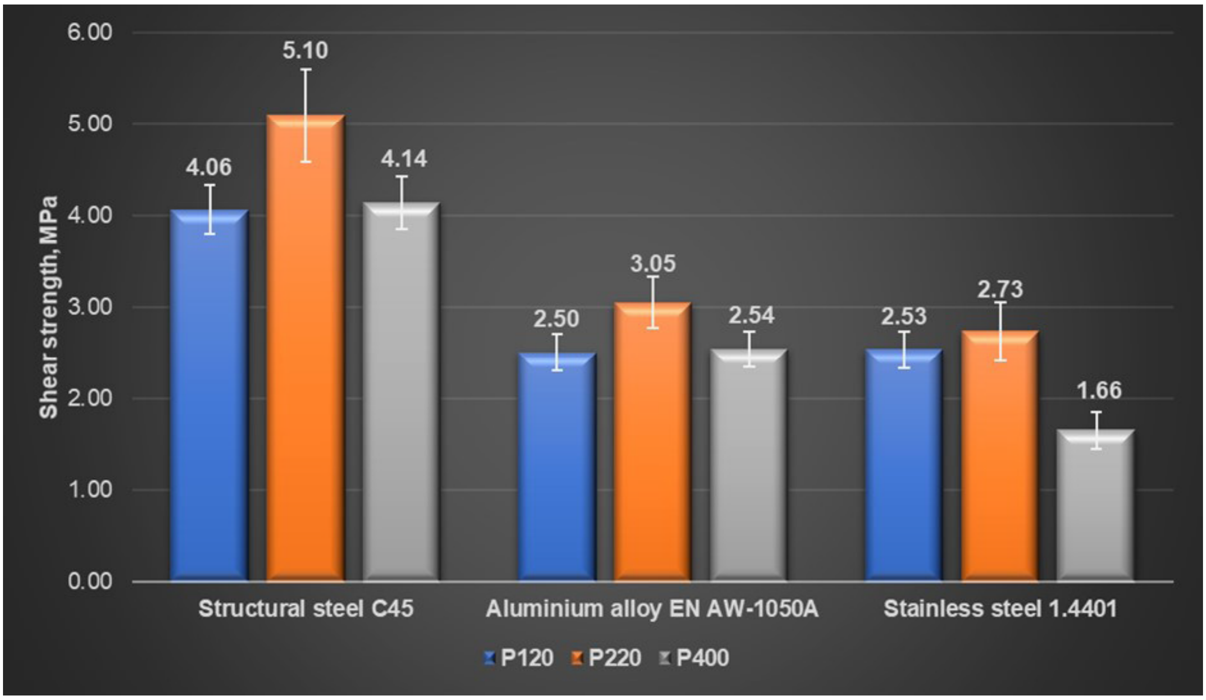

Fig. 2. Comparison of the shear strength results of adhesive joints made with E53/Z-1/100:10 due to the surface preparation method

Table 2. Results of the K-S distribution normality test

Type of material	Type of sanding paper	Level p for K-S test	Normality of distribution
Structural steel C45	P120	p > .20	YES
Structural steel C45	P220	p > .20	YES
Structural steel C45	P400	p > .20	YES
Aluminium alloy EN AW-1050A	P120	p > .20	YES
Aluminium alloy EN AW-1050A	P220	p > .20	YES
Aluminium alloy EN AW-1050A	P400	p > .20	YES
Stainless steel 1.4401	P120	p > .20	YES
Stainless steel 1.4401	P220	p > .20	YES
Stainless steel 1.4401	P400	p > .20	YES

Table 3. Levene's test results

	MS Effect	MS Error	The statistical value F	p level for the Levene test
Shear strength	0.024385	0.016731	1.457432	0.210450

Table 4. Results of Tukey's homogeneous post-hoc group test (HSD)

Type of material	Type of sanding paper	Shear strength	Homogenous group			
			1	2	3	4
Structural steel C45	P120	4.06		****		
Structural steel C45	P220	5.10				****
Structural steel C45	P400	4.14		****		
Aluminium alloy EN AW-1050A	P120	2.50	****			
Aluminium alloy EN AW-1050A	P220	3.05	****			
Aluminium alloy EN AW-1050A	P400	2.54	****			
Stainless steel 1.4401	P120	2.53	****			
Stainless steel 1.4401	P220	2.73	****			
Stainless steel 1.4401	P400	1.66			****	

accurate statistical analysis of the results obtained. Therefore, the normal distribution of the obtained results was checked at the beginning. For this purpose the Kolmogorov-Smirnov test (K-S) was used. The results of this test are presented in Table 2.

The results presented in Table 2 indicate that the distribution of the analyzed results is consistent with the normal distribution. Therefore, in the next step the assumption of equal variance was checked using Levene's test. The results of this test are presented in Table 3.

Level p for the Levene test is 0.211, which is higher than the assumed significance level of 0.05, which means that the assumption of the uniformity of variance is fulfilled. Thus, ANOVA was analyzed using the post-hoc test. The results of the homogeneous Tukey's test (HSD) are presented in Table 4.

The aim of the test was to determine which average values differ significantly and to separate groups of adhesive joints whose average shear strength values are at a similar level. Tukey's test formed 4 homogeneous groups. Analyzing the test results, it can be seen that the joints with the highest strength (made of sheets of C45 structural steel, the surfaces of which have been roughened with P220 abrasive paper are in a separate homogeneous group. Similarly, the joints which showed the lowest shear strength, i.e. the joints of stainless steel sheets 1.4401. This means that none of the other groups of joints had a similar strength result at the assumed level of materiality. In the case of adhesive joints of C45 structural steel sheets, the surfaces of which were prepared using P120 and P400 sandpaper, the average values are in one homogeneous group, i.e. with the assumed level of materiality α = 0.05 they do not differ significantly. The remaining groups of samples, which were analyzed during the work, i.e. adhesive joints made of aluminum alloy sheets EN AW-1050A, whose surfaces were prepared with abrasive papers of gradations P120, P220 and P400, as well as joints of stainless steel 1.4401, whose surfaces were prepared with abrasive papers P120 and P220, are in one homogeneous group, i.e. there are no significant differences between them at the assumed level of materiality.

Conclusions

On the basis of the presented results of the experimental research, it can be concluded that the selection of an appropriate method of surface preparation and appropriate tools for their implementation has a significant impact on the strength of the adhesive joints. In the case of mechanical processing with a coated abrasive tool, the proper selection of the abrasive tool gradation is of particular importance. The analysis of the conducted tests shows that in case of adhesive joining of selected structural materials the most advantageous results were obtained in case of joining sheets of C45 structural steel. This may be due to the properties of the material itself, as this steel has better workability compared to the aluminium alloy EN AW-1050A and stainless steel 1.4401. The best method of treatment for all materials used in the tests turned out to be surface treatment with abrasive grit P220. Adhesive joints made of metal sheets subjected to such treatment were characterized by the highest shear strength.

The lowest strength of all joints was found in the joints of 1.4401 stainless steel sheets, the surface of which was prepared using P400 graded abrasive paper. Such an effect may be due to the fact that this sheet is characterized by the highest hardness among the materials analyzed in the study (Table 1), and the use of fine grain abrasive did not give a sufficient surface development.

In summary, it should be stated that mechanical surface treatment of the materials to be joined has a significant impact on the strength of the adhesive joints. The use of abrasive paper with too large a grain size may result in too deep cavities where the adhesive may not reach, while in the case of the use of fine papers there is a risk of insufficient surface development. However, this is also strictly related to the properties of the material, especially its hardness, as well as the properties of the adhesive used, especially its viscosity. The application of an appropriate gradation of the abrasive allows to properly prepare the surface of the elements to be joined and, as a result, to obtain a strong adhesive bond, which is the result of a strong adhesion of the adhesive to the material surface.

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