PULL-OFF TEST OF ADHESIVE JOINTS BASED ON POLYESTER-GLASS LAMINATE AND ALUMINUM ALLOY

Badanie wytrzymałości na odrywanie połączeń klejowych przygotowanych na bazie laminatu poliestrowo-szklanego i stopu aluminium

Исследование прочности на отрыв клеевых соединений изготовленных из стеклянно-полестрого ламината и сплава алюминего

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DOI: 10.15199/160.2019.4.1

A b s t r a c t: Adhesive joints are widely used in various industries to join different materials. They always have to meet specific requirements for joint strength or durability. The article presents the results of research on the problem of joining elements made of aluminum alloy and polymer composite material present in the railway industry. The tested adhesive joints combine EN AW-5754 aluminum alloy (AlMg3) with a polyester-glass laminate. Three types of adhesives were used in the tests: epoxy, methacrylic and cyanoacrylate. Various methods of preparing the base surface and the bonded elements were used. The adhesive bonded surfaces were prepared using sandblasting and degreasing, grinding and degreasing as well as only degreasing. The research used a composite made with the use of two different technologies: contact molding and vacuum molding. Comparative tests were also carried out by making adhesive joints of two aluminum surfaces. The aim of the research was to propose a gluing technology to achieve min 15 MPa of the pull-off strength. The tests show that the required strength of the joint can be achieved only for an adhesive joint based on epoxy or methacrylic adhesive when preparing the surface of the base material by sanding and degreasing, grinding and degreasing, or even only degreasing.
K e y w o r d s : Adhesive joints, polyester-glass laminate, aluminum alloy, pull-off strength

S t r e s z c z e n i e: Połączenia klejowe są szeroko stosowane w różnych branżach przemysłowych do łączenia materiałów o odmiennych właściwościach mechanicznych i użytkowych. Zawsze muszą one spełniać określone wymagania w zakresie wytrzymałości czy też trwałości. W artykule zaprezentowano wyniki badań dotyczące problemu łączenia elementów wykonanych ze stopu aluminium i polimerowego materiału kompozytowego występującego w branży kolejowej. Badano połączenia adhezyjne występujące pomiędzy stopem aluminium EN AW-5754 (AlMg3) a laminatem poliestrowo-szklanym. W badaniach zastosowano trzy rodzaje klejów: epoksydowy, metakrylowy i cyjanoakrylowy. Zastosowano również różne sposoby przygotowania powierzchni do klejenia. Powierzchnie klejone przygotowano stosując piaskowanie i odtłuszczanie, szlifowanie i odtłuszczanie oraz odtłuszczanie. W badaniach zastosowano kompozyt wykonany dwoma różnymi technologiami: tzw. technologią na mokro (contact molding) oraz z wykorzystaniem worka próżniowego (vacuum molding). Przeprowadzono również badania porównawcze wykonując połączenia klejowe dwóch powierzchni aluminiowych. Celem badań było zaproponowanie technologii wykonania połączenia tak aby wytrzymałość doraźna połączeń na odrywanie była nie mniejsza niż 15 MPa. Z przeprowadzonych badań wynika, że przyjętą wytrzymałość połączenia można osiągnąć wykorzystując klej epoksydowy lub metakrylowy po przygotowaniu powierzchni materiału bazowego poprzez piaskowanie i odtłuszczanie, szlifowanie i odtłuszczanie lub odtłuszczanie.
S ł o w a k l u c z o w e : Połączenia adhezyjne, laminat poliestrowo-szklany, stop aluminium, wytrzymałość na odrywanie

Introduction

Adhesive joints are becoming more and more popular and they are a particularly attractive method of joining construction materials with different mechanical and functional properties, e.g. metal and composite elements [1, 2]. In work [4] a review of conditions generating the demand for adhesive joints was presented. In construction, adhesive joints are used, for example, to joint composite laminates with steel beams [8]. For joining metal and composite elements, adhesive joints are also used in aircraft constructions [5], in turbine production [10] or in pipe joints [11]. A special solution in which adhesive joints are used is the production of hybrid composites, in which metal layers, e.g. aluminum foils, are alternately joined with a composite material to form a laminate with unique properties [9].

Previous studies of adhesive joints based on polyester-glass laminates and aluminum show that in addition to the identified problems defined for adhesive joints, there are new, associated with limited interlayer strength of the laminates. A non-uniform distribution of stress in the adhesive joints and stress concentration effects at the ends of their overlap cause the pull-off and peeling phenomena not only in the adhesive, but also in the joined composite element. Consequently, as a result of the joint load, not only the joint is damaged, but very often also the composite material as a result of exceeding the adhesive interlayer strength of the laminate. These types of problems do not only occur when joining metal parts [3, 6, 7].

This paper presents the results of experimental tests of adhesive-bonded tensile joints used for joining elements made of aluminum alloy and polymer composite material.

Research problem, assumptions and research goal

The problem considered in this paper concerns the production of adhesive joints for use in the railway industry. The analyzed joints is used in the process of mounting the locomotive lighting housing to the locomotive cabin. The material used in the production of the locomotive body is a polymer composite. The lighting housing is an aluminum element. The problem concerns the selection of appropriate materials and conditions for the production of the adhesive joint to obtain a joint with a minimum pull-off strength of 15 MPa. In the work various types of adhesives (epoxy – EP, methacrylic – MK, cyanoacrylic – CA), various methods of surface preparation (sandblasting and degreasing – PO, grinding and degreasing – SO, degreasing – O) and various methods of producing composite material (so-called wet technique – contact molding and the technique uses vacuum bag technology – vacuum molding) are used. In addition, comparative studies were carried out in which the base material was aluminum alloy. The aim of the study was to determine the pull-off strength of the adhesive joints.

Research plan

The research identified three factors that have a potential impact on the quality of adhesive joints: the materials to be joined, the type of adhesive, the method of preparing the surface of the base material (PP) and the element to be joint (PE). The tested materials are aluminum alloy EN AW-5754 (AlMg3), polyester-glass laminate made by hand technology (laminate A) and polyester-glass laminate made in vacuum technology (laminate B). The curing time for the adhesive bond was

Tab. 1. List of completed experimental tests; PO – Sandblasting and degreasing, SO – Grinding and degreasing, O – Degreasing, EP – Epoxy glue, MK – Methacrylate glue, CA – Cyanoacrylate glue
Tab.1. Lista zrealizowanych testów eksperymentalnych; PO – Piaskowanie i odtłuszczanie, SO – Szlifowanie i odtłuszczanie, O – Odtłuszczanie, EP – klej Epoksydowy, MK – klej Metakrylowy, CA – klej Cyjanoakrylowy

Experiment No	Adhesive	Base material	Preparation of base material surface (BM)	Preparation of stamps surface (PE)	Curing time [hrs.]
E 1	EP	Aluminum	PO	PO	72
E 2	EP	Laminate A	SO	PO	72
E 3	EP	Laminate B	SO	PO	72
E 4	MK	Aluminum	PO	PO	24
E 5	MK	Laminate A	SO	PO	24
E 6	MK	Laminate B	SO	PO	24
E 7	CA	Aluminum	PO	PO	24
E 8	CA	Laminate A	SO	PO	24
E 9	CA	Laminate B	SO	PO	24
E 10	EP	Aluminum	SO	SO	72
E 11	EP	Laminate A	SO	SO	72
E 12	EP	Laminate B	SO	SO	72
E 13	MK	Aluminum	SO	SO	24
E 14	MK	Laminate A	SO	SO	24
E 15	MK	Laminate B	SO	SO	24
E 16	CA	Aluminum	SO	SO	24
E 17	CA	Laminate A	SO	SO	24
E 18	CA	Laminate B	SO	SO	24
E 19	EP	Aluminum	O	O	72
E 20	EP	Laminate A	O	O	72
E 21	EP	Laminate B	O	O	72
E 22	MK	Aluminum	O	O	24
E 23	MK	Laminate A	O	O	24
E 24	MK	Laminate B	O	O	24
E 25	CA	Aluminum	O	O	24
E 26	CA	Laminate A	O	O	24
E 27	CA	Laminate B	O	O	24

Tab. 2. The applied adhesive curing conditions
Tab.2. Zastosowane warunki utwardzania kleju

Adhesive	Temperature [°C]	Lifetime of adhesive	Stabilization (immobilization) time	Full curing time
Epoxy	23	40 min	4 hrs.	72 hrs.
Methacrylic	23	10 min	20 min	24 hrs.
Cyanoacrylic	23	60 sec	5 min	24 hrs.

24 or 72 hours. 27 experimental tests were carried out (Table 1). In each test, two plates of material in a form of A4 and 10 aluminum stamps bonded to the plates were used, 5 pieces per plate.

Experimental tests were carried out in series of three, which resulted from the number of solutions in the frame of preparing the surface for jointing. The adhesive used in the tests had the same production series.

Specimens preparation

In order to remove impurities, three types of surface preparation for adhesive bonding were used in the tests: degreasing, sandblasting and grinding. Degreasing was carried out immediately before grinding, sandblasting and adhesive processes. The material was degreased with an organic solvent (Isopropanol). After degreasing, the material was allowed to evaporate for 10 minutes. Surface treatment and adhesive application was carried out within 20 minutes after degreasing. Sandblasting was done manually in a pressure sandblaster. Corundum abrasive F46 was used for sandblasting, the jet pressure did not exceed 8 bar. The sandblasting head was held at an angle of 45-60 degrees, the head was moved smoothly and uniformly. Sandblasting time was chosen so as to obtain a matt surface, without gloss or discoloration. Properly processed material has the same tone over the entire surface used. Grinding was done manually with a belt grinder. cross grinded technique was applied. The first grinding was done to remove the surface layer and impurities. The second grinding, transverse to the first, was to remove traces of the previous one and results in obtaining an even surface roughness. Abrasive paper with P60 gradation was used. After machining, the surfaces were washed again with solvent.

Used materials

The applied epoxy adhesive is a two component chemically cured adhesive. The curing process was initiated by mixing two components (resin and hardener) at room temperature. It was a polyaddition adhesive, which means that resin and hardener alternate in the polymer chain of such an adhesive, so it requires a large amount of hardener compared to other adhesives. The second adhesive used was methacrylic adhesive. It is also a two-component chemically cured adhesive. The curing process was initiated by mixing the two components at room temperature. Like the epoxy adhesive, it cured to duromer form and had similar mechanical and performance properties. The difference between these adhesives is due to the mechanism of polymer chain formation. Methacric adhesive belongs to polymerization adhesives. This type of chemical reaction does not require alternating hardener and resin chain order. This adhesive cures more easily and is more tolerant to impurities or improper surface preparation. The last used adhesive, cyanoacrylate adhesive, is a one-component adhesive. The hardener is water in the form of moisture on the surfaces to be joined. The curing process begins when it touches the surface. The recommended relative humidity is between 30 and 60%. This is called instant adhesive cured in a few seconds. A special type of adhesive with a slow curing process was used for the test to extend the lifetime of adhesive. Only the adhesive that has direct contact with moisture cures on the joined surfaces, therefore the maximum joint thickness is 0.2 millimeters. The cured adhesive is in the form of a thermoplastic material – a plastic that is sensitive to temperature, solvents and moisture. The applied adhesive curing conditions are shown in Table 2.

Two laminates were used in the joints. Laminate A is a glass-polyester laminate made by hand (called hand lay-up), which involves manual supersaturation of polyester resin of glass fabrics arranged in the form of intersecting layers. Laminate B is a glass-polyester laminate made by vacuum forming. The method of implementation consists in sucking air from the mold while saturating glass fabrics with resin. This ensures much more accurate resin impregnation, eliminates air bubbles and positively affects the homogeneity of the laminate. In the study A4 panels cut from panels of larger sizes were used. This way, the "edge" part of the material with the largest number of imperfections was eliminated. The panels were coated with gelcoat used to produce the locomotive cabin. In the research aluminum alloy EN AW-5754 (AlMg3) in the form of A4 sheets with a thickness of 2 mm was additionally used.

Conditions for making adhesive joints

The bonding process took place in the adhesive zone prepared in accordance with DIN 6701, the so-called "Clean room". Conditions prevailing during the making of adhesive joints and during the curing of the adhesive were regulated and monitored to ensure their repeatability. Due

Fig. 1. Removing adhesive outflows
Rys. 1. Usuwanie nadmiaru kleju

Fig. 2. Pull-off testing
Rys. 2. Zrywanie próbki

to the influence of temperature on the curing time of the adhesive and the final strength of the adhesive joint, the temperature was maintained in the range of 22 to 24 degrees Celsius during the process. The relative humidity in the room ranged from 30 to 60%. All materials used in the study (metals, laminates, adhesives, solvents) were subject to a 24-hour acclimatization process in the adhesive zone. The exception were sandblasted elements, which had to be bonded within 24 hours of sandblasting and were acclimatized before sandblasting. No substances (silicone adhesives, greases, latex gloves, hand protection creams) containing silicone, which is an anti-adhesive agent, did not get into the adhesive area. Powder-free nitrile gloves were used when making adhesive joints.

The method of assessment of pull-off strength of the adhesive joints

The pull-off strength test was carried out with the use of pull-off method, belonging to destructive tests. It allows, among others, to determine the adhesion forces occurring between the joined materials, including pull-off strength. The device used for testing is the PosiTest AT-A Automatic Adhesion Tester. The meter measures the adhesion forces by pull-off a stamp jointed to the material, using a hydraulic actuator and a smoothly increased force. The mechanism of the device is based on the use of ball bearings in the mounting ring, which ensures a perpendicular distribution of forces on the pull-off surface.

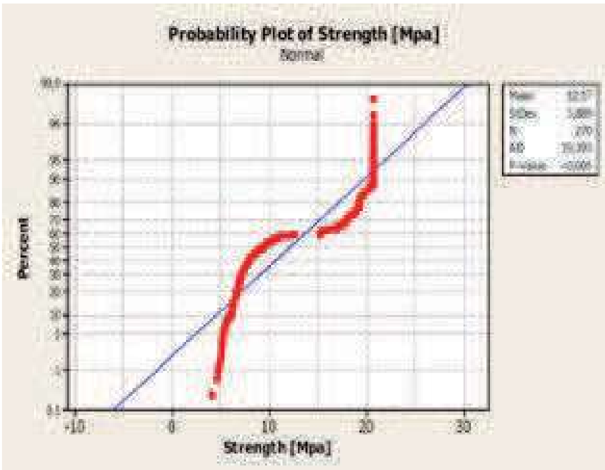
The test used stamps with a diameter of 20 mm dedicated to the measuring range of 0-20 MPa. The device resolution is 0.01 MPa. The force measurement result is given on the electronic display in MPa or psi with an accuracy of ±1%. Before testing, adhesive outflows were removed from the samples with a suitable knife (Fig. 1). Figure 2 shows an example of a sample pull-off testing.

Research results and their analysis

The results of the tests carried out are presented in Table 3 presenting information on the average,

Fig. 3. Adhesive destruction of gelcoat layer – experiment E12
Rys. 3. Zniszczenie adhezyjne warstwy żelkotu – eksperyment E12

Fig. 4. Normality test results of the di stribution of strength data
Rys. 4. Ocena normalności rozkładu danych dotyczących wytrzymałości



Tab. 3. Experimental tests results
Tab. 3. Wyniki badań eksperymentalnych

Exper.	Pull-off strength [MPa]				The type of destruction
	Mean	Max	Min	St. Dev.	
E 1	20.68*	-	-	-	-
E 2	7.11	7.67	6.75	0.31	Cohesive of material
E 3	19.51	20.68	17.16	1.18	Cohesive of material
E 4	20.68*	-	-	-	-
E 5	7.04	7.78	6.32	0.42	Cohesive of material
E 6	18.87	20.68	17.76	1.02	Cohesive of material
E 7	15.39	15.93	15.18	0.22	Adhesive of gelcoat layers
E 8	7.43	7.95	6.97	0.31	Cohesive of material
E 9	11.04	12.58	9.42	0.88	Cohesive of material
E 10	19.32	19.5	19.18	0.12	Adhesive mixed of joint
E 11	6.926	8.03	6.03	0.72	Cohesive of material
E 12	17.98	18.95	16.8	0.78	Cohesive of material
E 13	20.68*	-	-	-	-
E 14	7.53	8.04	6.81	0.50	Cohesive of material
E 15	18.81	19.65	18.03	0.60	Cohesive of material
E 16	8.93	9.52	7.94	0.47	Adhesive of gelcoat layers
E 17	6.71	7.91	6.12	0.53	Cohesive of material
E 18	8.17	8.54	7.56	0.34	Adhesive of gelcoat layers
E 19	5.36	6.26	4.9	0.39	Adhesive mixed of joint
E 20	5.28	6.5	4.14	0.69	Mixed cohesive / adhesive of joint in the ratio 80/20 (plate 1), 70/30 (plate 2)
E 21	5.69	6.58	5.1	0.45	Adhesive mixed of joint
E 22	17.20	17.85	16.08	0.57	Adhesive mixed of joint
E 23	6.84	7.26	6.15	0.31	Cohesive of material
E 24	19.56	20.33	18.93	0.47	Mixed cohesive / adhesive of joint in the ratio 90/10
E 25	10.17	10.71	9.39	0.46	Adhesive mixed of joint
E 26	6.54	7.53	6.22	0.40	Cohesive of material
E 27	9.13	9.94	8.32	0.57	Mixed cohesive / adhesive of joint in the ratio 80/20

maximum and minimum pull-off strength obtained in the experiments, standard deviation and information on the type of joints destruction.

The data presented in Table 3 show that various forms of damage occurred not only in the joint, but also in the composite material (KM) itself, adhesive of gelcoat layer (AP) (Fig. 3), mixed cohesive / adhesive of joints (KAM) and mixed adhesive of joints (AM). The presented results were further analyzed. First, the normality test of the distribution of the obtained strength values was made. From the results obtained (Fig. 4) it can be concluded that the distribution is not normal (P-value <0.05).

Because the data distribution is not normal, the Kruskal-Wallis test was used to identify factors with a statistically justified effect on strength. A confidence level of 95% was adopted. Tests were carried out using the Minitab 16. Based on the obtained P-values, conclusions were drawn regarding which of the following factors have a statistically justified effect on the strength of the adhesive joint: type of adhesive (P-value = 0.000), type of base material (P-value = 0.035), method of preparing the surface of the stamp (P-value = 0.000), surface preparation method for the base material (P-value = 0.000). The tests show that all variables have a statistically justified effect on the obtained strength values. Therefore, further analyzes were carried out to define the nature of this impact. The analyzes were carried out using the Minitab 16 and Statistica 12. First, the analysis of the impact of the type of base material on the strength of the adhesive joint was performed. The obtained results are shown in Fig. 5. In the case of Laminate A, the required strength of at least 15 MPa was not obtained. Therefore, in further analyzes the joints made with the use of this material was not included. Fig. 6 shows the obtained average strength values for various types of adhesives. The highest average strength was obtained for methacrylic glue (MK).

Fig. 5. Joint strength for various base materials (BM)
Rys. 5. Wytrzymałość połączenia dla różnych materiałów bazowych (BM)

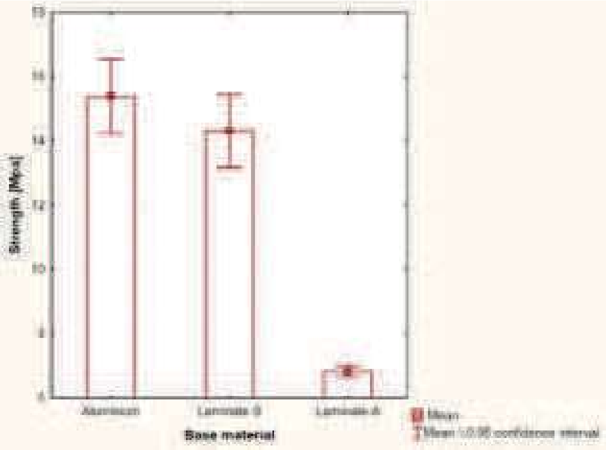


Fig. 6. Joint strength for various types of adhesives
Rys. 6. Wytrzymałość połączenia dla różnych rodzajów klejów

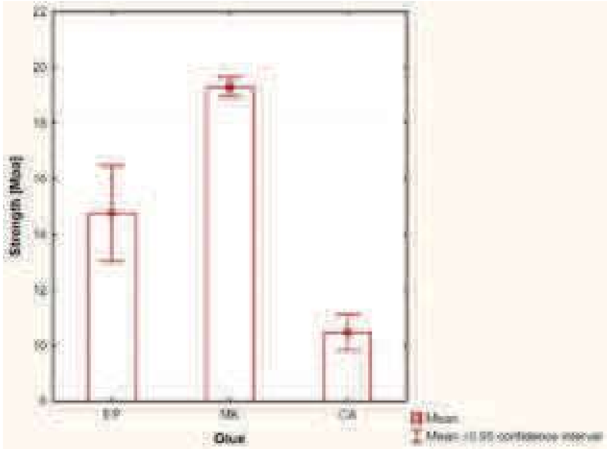


Fig. 7. Joint strength for various methods of surface preparation of base materials (BM)
Rys. 7. Wytrzymałość połączeń dla różnych sposobów przygotowania powierzchni materiałów bazowych (BM)

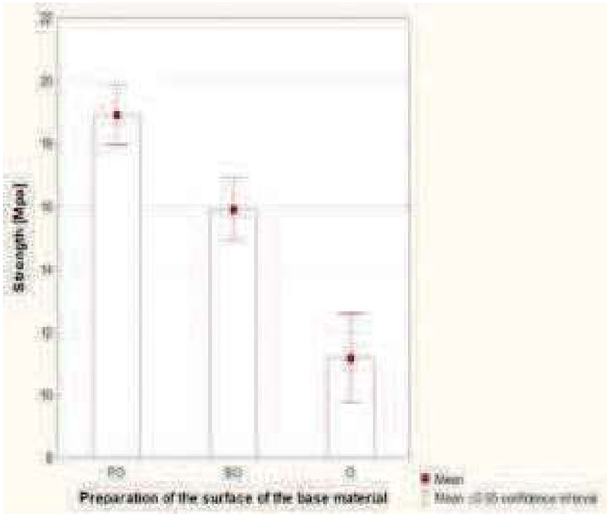


Fig. 8. Joint strength for various methods of surface preparation of joined elements (stamps) (PE)
Rys. 8. Wytrzymałość połączeń dla różnych sposobów przygotowania powierzchni stempli (PE)

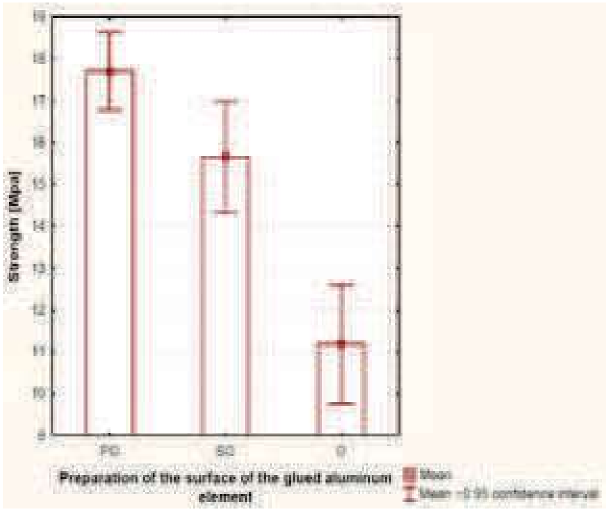


Fig. 9. Joint strength for various adhesives and base materials
Rys. 9. Wytrzymałość połączeń dla różnych klejów oraz materiałów bazowych

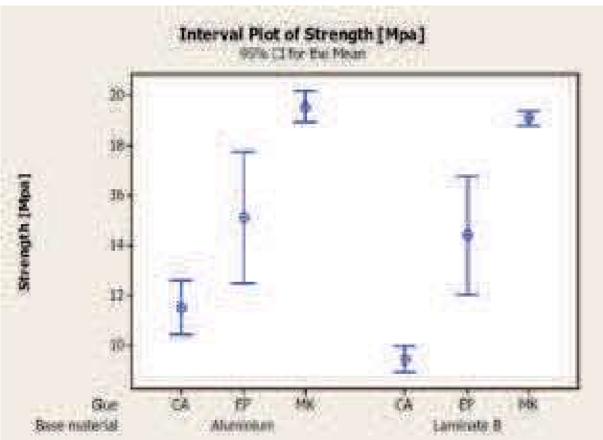
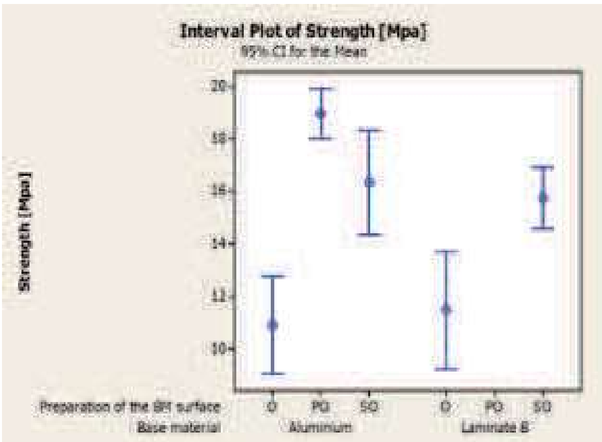


Fig. 10. Joint strength for different ways of the base material surface preparation and various base materials
Rys. 10. Wytrzymałość połączeń dla różnych sposobów przygotowania powierzchni materiału bazowego oraz różnych materiałów bazowych



Tab. 4. List of adhesive joints meeting the condition that strength > 15 MPa
Tab. 4. Wykaz połączeń klejowych spełniających warunek, że wytrzymałość > 15 MPa

Experiment No	Adhesive	Base material	Preparation of base material surface (BM)	Preparation of stamps surface (PE)	Curing time [hrs.]	Mean strength [MPa]
E 10	EP	Aluminium	SO	SO	72	19,32
E 22	MK	Aluminium	O	O	24	17,20
E 7	CA	Aluminium	PO	PO	24	15,39
E 3	EP	Laminat B	SO	PO	72	19,51
E 12	EP	Laminat B	SO	SO	72	17,98
E 24	MK	Laminat B	O	O	24	19,56
E 6	MK	Laminat B	SO	PO	24	18,87
E 15	MK	Laminat B	SO	SO	24	18,81

In terms of the base surface preparation method, the best results were obtained for surfaces after sandblasting and degreasing (PO) (Fig. 7). All adhesive joints in which the surface of the base material was subjected to sandblasting and degreasing met the assumed condition. Assessing the method of preparation of the surface of the adherent element (stamp), the best results were also obtained for surfaces after sandblasting and degreasing (PO) (Fig. 8), although the assumed strength of joints was obtained for all types of preparation of the surface of the adhesive elements.

The strength for various adhesives as well as different ways of base material surface preparation for different base materials are presented in Figure 9 and Figure 10. The best results were obtained for Laminate B when using methacrylic adhesive and when grinding and degreasing of the glued surfaces were applied.

However, because the main purpose of this work was to indicate the conditions for preparing joints ensuring obtaining a pull-off strength of at least 15 MPa, an analysis of the results of individual experiments was conducted and Table 4 presents a list of tests in which this condition was met.

Summary

Based on the tests carried out, it can be concluded that the strength of the adhesive joint at a level of min 15 MPa was achieved only for Laminate B made using a vacuum bag technology. The required pull-off strength can already be achieved by only degreasing the base material surface and the adherent element (stamp), provided however that methacrylic adhesive is used. From the point of view of the labor consumption of the production process, using methacrylic adhesive, which does not require a complex process of preparing the surface for bonding, is justified. In solutions where methacrylic adhesive was used, grinding and sanding of the surface even deteriorated the pull-off strength of the joints.

In the destructive tests of adhesion of composite materials, there may be a problem of degradation not only of joints but also of the material being joined. This is because very often the adhesive strength of protective layers of composite materials, e.g. gelcoat, is less than the adhesive strength of an adhesive joint. Therefore, during the process of preparing the surface for bonding, it is necessary to consider the need to extend the preparatory procedure by mechanical removal of the protective layer – as in the case of adhesive bonding metal elements, the varnish layer is removed.

The suitability of using methacrylic adhesive in the process of mounting the locomotive lighting housing to its cabin should be verified in durability tests. In the case of adhesive joints, the temporary strength of the joint is an important parameter, but taking into account the aging processes occurring in structural adhesives, temporary tests are very often supplemented with durability tests.

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