

## Influence of Adhesive Compound Viscosity on the Strength Properties of 1.0503 Steel Sheets Adhesive Joints

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### ABSTRACT

The presented study concerned the comparison of the adhesive joints strengths of 1.0503 carbon steel sheets of higher quality, which were made using adhesive compounds characterized by different viscosity index of the components. Three types of commercial epoxy resins were used: Epidian 5, Epidian 53 and Epidian 57, as well as two types of curing agents: polyamide (PAC) and amine (Z-1). The surfaces of the test specimens were subjected to a pre-treatment operation in the process of a mechanical machining with an abrasive coated tool of P320 gradation. The surface roughness measurements were carried out as a control. The single-lap adhesive joints were the subject of the strength testing. The strength tests carried out on a Zwick/Roell Z150 testing machine concerned the comparison of the strength of the adhesive joints loaded in shear. Statistical analysis was performed on the results obtained. It was observed, among others, that the adhesive joints prepared with the epoxy adhesive compounds containing the polyamide curing agent showed much higher the shear strength than the adhesive joints made with the epoxy adhesive compounds containing the amine curing agent. Moreover, the use of the polyamide curing agent in the adhesive compounds with the epoxy resins resulted in formation of a more elastic adhesive layer, for which higher elongation was observed than in the case of the epoxy compound with the amine curing agent.

**Keywords:** adhesive joints, carbon steel, shear strength, epoxy adhesives, viscosity.

### INTRODUCTION

The bonding process makes it possible to prepare the assembly joints characterized by the strength properties similar to those of other joining methods, such as soldering, pressure welding or welding [1]. Therefore, the last few years have seen a dynamic development of adhesive technology, which is proving to be a promising the assembly technology due to the relatively low costs, reduced labour intensity and wide access to even specialize adhesive substances. In addition to the bonding function, the adhesive joints can also fulfil other functions such as sealing, regeneration or protection. Due to all the aspects discussed, the adhesive technology is used in practically all sectors of industry, both in aviation, automotive, construction, electrical engineering, as well as in light industry [2–4]. The

strength of the adhesive joints depends on many factors, such as physical and chemical phenomena (wettability, adhesion and cohesion), technological factors (the method of preparing the jointed materials' surfaces, the adhesive properties and the curing conditions of the adhesive joint and seasoning) [5–8] and construction factors (the geometry of the adhesive joint and the loading method) [4,9], as well as the operating conditions affecting the adhesive joints during operation [9–12]. One of the most popular construction adhesives are the epoxy adhesives [13, 14]. The epoxy adhesives are characterized by high load and tensile strength as well as high chemical and temperature resistance [15–17]. Taking into account the wide choice of both commercial resins and curing agents for preparing the epoxy adhesives, it is possible to obtain the most suitable product for the given working

conditions [17, 18]. The epoxy adhesives can be subjected to numerous modifications, using various types of the modifiers [18–20], as well as other polymers or the resins [21–26]. Considering the technology of joining, it is worth remembering that the phenomenon of adhesion is an important part of the process of forming constructional adhesive joints, affecting the strength of the joints. Adhesion is a phenomenon of the surface binding of two different objects together, under the influence of forces of attraction between them [2, 3, 27]. Different theories of adhesion are available: mechanical, electrostatic, diffusion, physico-chemical. From a structural point of view, the greatest contribution is attributed to the mechanical theory, which describes the influence of the surface condition on the quality of the bond. An extremely developed surface has many irregularities-crevices into which the adhesive substance may penetrate, therefore, according to this theory, one of the basic factors influencing the quality of the adhesive bond is the preparation of both adhesive surfaces and the selection of an appropriate adhesive [5, 28–30]. On the extended surface topography, there is a natural “interlocking” of the adhesive with the material surface, as the liquid adhesive fills in the irregularities on the material surface before curing. However, as the authors Kaczorowska and Batista presented in their works [31, 32] not in every case a more developed surface works in the advantage of the joint, as a substance with high viscosity will not fill small, numerous cavities. Therefore, when preparing an adhesive joint, an adhesive with the appropriate viscosity should be selected with attention to the type of material to be bonded and the method of surface preparation. In this study the influence of the viscosity of the adhesive compounds on the strength properties of 1.0503 steel adhesive joints was determined. The adhesive compounds consisting of three epoxy resins and two curing agents of different viscosities were analysed. The tests were carried out on single-layer sheet metal adhesive joints, the surfaces of which were treated mechanically with an abrasive coated tool, and then degreased with technical acetone.

## METHODS OF RESEARCH

### Adherends used in the studies and surface treatment

The specimens used for the test were cuboid specimens of 100 × 25 × 2 mm cut from a sheet of 1.0503 carbon steel. This material is a high quality, non-alloyed steel [33]. Table 1 shows the chemical composition of 1.0503 steel. Table 2 presents the basic mechanical properties of the 1.0503 steel used in the tests. This steel is widely available and, due to its properties, is used in the production of machine parts, tools and mounting plates. 1.0503 steel is easy to machine, so it is available in many forms, including sheet metal, pipes and bars in various shapes, but it is difficult to weld. The surface of the samples to be bonded was subjected to mechanical abrasive treatment to order to develop it properly. The surface treatment process was carried out in several stages:

1. Degreasing the specimens with technical acetone to remove grease, dust and contamination left over from the cutting process.
2. Dry abrasive machining using a P320 gradation corundum coated abrasive tool. The machining was carried out using a BOSCH PSS 250AE oscillating grinder. The surface was treated for 1 minute.
3. Degreasing the treated surface to remove dust, dirt and residues using technical acetone.

Before the bonding, the quality of the prepared surface was checked by the surface roughness tests.

**Table 2.** Basic mechanical properties of the 1.0503 steel [33, 34]

Mechanical properties	1.0503 steel
Tensile strength Rm	560–850 MPa
Elongation	14%–17%
Narrowing	35%–45%
Yield strength Re	275–490 MPa
Hardness after softening	≤ 229 HB
Breaking work	≥ 25 J

**Table 1.** Chemical composition of 1.0503 steel used in the tests [33, 34]

Element	C	Si	Mn	Cr	Ni	Mo	Cu	S	P
Contents [%]	0.42–0.50	0.1–0.4	0.5–0.8	max 0.3	max 0.3	max 0.1	max 0.3	max 0.04	max 0.04

### Adhesives used in the tests

Six types of two-component epoxy adhesives were used when making the adhesive joints. Three types of the epoxy resins of different viscosity were used: Epidian 5, Epidian 53 and Epidian 57. Each resin was mixed with two types of hardeners, also differing significantly in viscosity: PAC curing agent and Z-1 curing agent in manufacturer-specified proportions. All components of the adhesives used in the study were manufactured by the Polish company CIECH S.A (Ciech Sarzyna S.A., Nowa Sarzyna, Poland).

Epidian 5 epoxy resin is a pure form resin produced as a reaction product of bisphenol A with epichlorohydrin. Epidian 5 epoxy resin is characterised by excellent adhesion to most types of plastics. It is also characterised by chemical resistance, as well as resistance to aggressive environmental factors. It is also distinguished by good electrical properties [26, 35, 36]. Compositions based on epoxy resin Epidian 5 are used to produce glass fibre laminates and joining various materials such as metals, ceramics and thermosetting plastics. Epidian 5 epoxy resin adhesive compositions are applied also in building structures as anti-corrosive or electro-insulating coatings. Epidian 53 epoxy resin is a mixture of epoxy resin obtained from bisphenol A and epichlorohydrin with average molecular weight  $\leq 700$  and styrene. Epidian 53 resin has high shear strength at temperature about 110°C [26, 36, 37]. This epoxy resin is used in joining glass laminates. Due to its excellent electrical insulating and strength properties, it can be used in radio technology, aviation and optics. Epidian 57 epoxy resin is a mixture of epoxy resin obtained from bisphenol A and epichlorohydrin with the average molecular mass  $\leq 700$  and thinner (saturated polyester resin). Its primary use is as a basic component

in adhesives for bonding metals, glass, ceramics and wood [26, 36]. Table 3 summarises the physical and chemical properties of the described epoxy resins. The curing agent PAC, consisting of fatty acids and polymeric reaction products with triethylenetetramine, is used to cure liquid epoxy resins. It increases the flexibility and impact strength of the compound, therefore it is used for deformation prone joints, e.g. in boat-building for joining wooden elements or elements made of polyester-glass laminate, for joining rubber with metal, thin metal sheets, plywood, and for pouring of elements in electrical engineering and electronics. PAC curing agent belongs to the group of slow reacting hardeners and its use time is 180 minutes at room temperature. This is followed by an initial cure of 6–8 hours to achieve an approximate 80–90% cure after 72 hours. Complete cure is achieved after 7–14 days. This process can be accelerated by using a higher temperature after the first stage of curing. The Z-1 curing agent (aliphatic amine) is mainly used in compounds with low molecular weight epoxy resins and products based on epoxy resins. When the curing agent is added to the resin, the curing process begins, and a certain amount of time is allowed for the mixture to be used before it is cured (open time). This time is dependent on several factors: temperature, amount of mixture, etc. and will vary with individual conditions. The gel time is approximately 35 minutes at room temperature. Initial cure is achieved after 48 hours - cure rate is approximately 80–90% and full cure after from 7 to 14 days.

Table 4 summarises the performance properties of the curing agents used in the tests. The compounds used in the study are presented in Table 5. The adhesive compound was prepared immediately before the bonding process. The components

**Table 3.** Functional properties of the epoxy resins used in the study [26, 35–37]

Property	Epidian 5 epoxy resin	Epidian 53 epoxy resin	Epidian 57 epoxy resin
Epoxy number	0.48–0.52 mol/100 g	$\geq 0.41$ mol/100 g	$\geq 0.40$ mol/100 g
pH value	approx. 7	approx. 7	approx. 7
Viscosity at 25°C	20 000–30 000 mPa·s	900–1 500 mPa·s	13 000–19 000 mPa·s
Density at 20°C	1.16 g/cm <sup>3</sup>	1.11–1.15 g/cm <sup>3</sup>	1.14–1.17 g/cm <sup>3</sup>
Flash point	266°C	75°C	134°C
Self-ignition temperature	490°C	460°C	455°C
Melting point	30–50 °C	not applicable	Not applicable
Boiling point	not indicated - decomposition	141°C	> 215°C does not boil over

**Table 4.** Properties of the described curing agents [36, 38]

Property	PAC curing agent	Z-1 curing agent
Viscosity at 25°C	10 000–25 000 mPa·s	20–30 mPa·s
Density at 20°C	1.10–1.20 g/cm <sup>3</sup>	0.978–0.983 g/cm <sup>3</sup>
Amine number	290–360 mg KOH/g	min. 1 100 mg KOH/g
Gelling time (e.g. for compounds with Epidian 5 at 20°C, for a 100 g sample)	180 min	33 min

**Table 5.** Types of epoxy adhesives used in testing

Type of resin	Type of curing agent	Resin/curing agent ratio by weight	Adhesive designation
Epidian 5	PAC curing agent	100:80	E5/PAC/100:80
Epidian 53			E53/PAC/100:80
Epidian 57			E57/PAC/100:80
Epidian 5	Z-1 curing agent	100:12	E5/Z-1/100:12
Epidian 53			E53/Z-1/100:10
Epidian 57		100:10	E57/Z-1/100:10

of the mixtures were accurately weighed using a KERN CKE 3600-2 laboratory scale with a measurement accuracy of 0.01 g. They were then mixed using a mechanical mixer marked Güde GTB 16/5 A fitted with a propeller agitator. The speed of the mixer was 460 rpm and the mixing process took 2 min. Then the adhesive compounds were degassed for 2 min.

### Adhesive joints

The single-lap adhesive joints with an overlap length of  $l_z = 18$  mm were tested. The length of the adhesive joint overlap was determined from the relation presented in papers [39, 40], allowing the calculation of the limiting length of the overlap in the case of the joining materials of equal thicknesses. The prepared adhesive compounds were applied to the bonded surfaces with a brush to apply the adhesive, allowing a homogeneous joint thickness to be achieved over the entire bonding surface. The next step was to join the parts. The joints were subjected to a one-stage curing process at room temperature with a load of 1 kg. Total curing time was 7 days. For each type of adhesive, 10 adhesive joints were prepared. The entire adhesive bonding process, including surface treatment, was conducted under laboratory conditions at  $22 \pm 1^\circ\text{C}$  and 30% air humidity. After the curing time and before testing the adhesive bonds, the adhesive flashes formed during the bonding process were removed.

### Test stations

#### Surface roughness tests

Surface roughness tests were carried out using a T8000 RC120-400 contour, roughness and 3D topography measuring device from Hommel-Etamic. Testing was carried out in accordance with PN-EN ISO 25178 standard [41]. A TKU300 measuring tip was used in the study. The measuring range was 80  $\mu\text{m}$ . The mapping distance was 4.80 mm and the tests were carried out 10 times on the surface of one sample. Tests were carried out at a speed of 0.80 mm/s.

#### Strength tests

When the assumed curing time had elapsed, all adhesive bonds were subjected to destructive endurance testing on a Zwick/Roell Z150 testing machine. Determination of the shear strength. These tests were conducted in accordance with the PN-EN 1465:2009 standard [42]. The crosshead speed during the test was 5 mm/min with a pre-test force of 5 N.

## RESULTS

### Surface roughness and topography tests

The surface roughness tests were carried out on 6 randomly selected samples, whose surface were subjected to the mechanical treatment (abrasive) and for comparison the parameters

of a reference sample, whose surface was only degreased, were measured. The following roughness profile parameters were determined during the test (PN-EN ISO 25178 standard):

- $Rt$  – total height of the profile,
- $Ra$  – arithmetic mean deviation of the roughness profile,
- $Rq$  – mean square deviation of the profile from the mean line along the measuring or elementary section,
- $Rz$  – height of roughness according to ten points of the profile,
- $Rp$  – height of the highest elevation of the profile within an elementary section,
- $RSm$  – mean width of the profile grooves.

The average results obtained for each parameter are summarised in Table 6. Figure 1 shows the surface roughness profiles of the tested samples.

Analysing the obtained results of the roughness parameters and the obtained roughness profiles, one may notice that there was a significant development of the structure of the surface

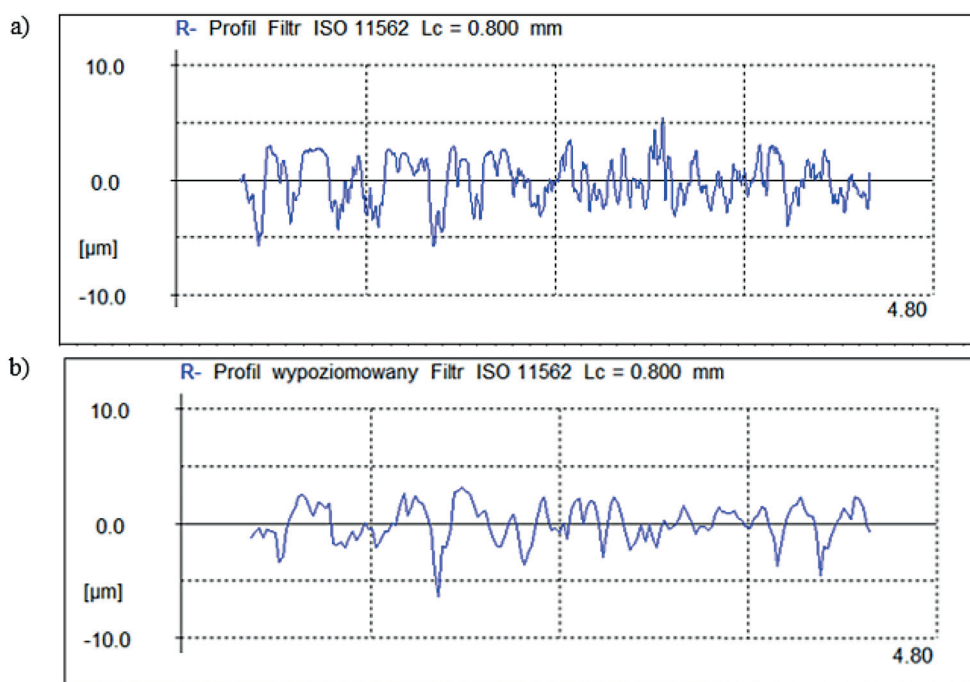
intended for the adhesive process because of the performed mechanical treatment. The noticeable widening of the width of the profile grooves and deviations from the 0.0 line on the profile of the sample subjected to the abrasive treatment testify to the occurrence of the irregularities in which the adhesive will have the possibility to “anchor” according to the mechanical theory of adhesion.

### Strength test results

Figure 2 shows the results of the strength tests of the adhesive joints analysed in this study. Analysing the found test results presented, it can be seen that the adhesive joints made with the use of the epoxy compounds containing PAC curing agent are much stronger than the joints made with the use of the epoxy compounds containing Z-1 curing agent. The highest strength was obtained for the adhesive joints prepared with the use of a compound consisting of the Epidian 53 epoxy resin and PAC curing agent (14.93 MPa),

**Table 6.** Averaged roughness parameters of tested samples

Specimen	Surface roughness parameters [ $\mu\text{m}$ ]					
	$Rt$	$Ra$	$Rq$	$Rz$	$Rp$	$RSm$
Reference	6.87	0.74	1.34	3.70	2.14	0.11
After abrasive treatment	8.45	1.11	2.14	5.16	2.23	0.22



**Fig. 1.** Roughness profile of test samples: a) reference sample, b) sample after abrasive treatment

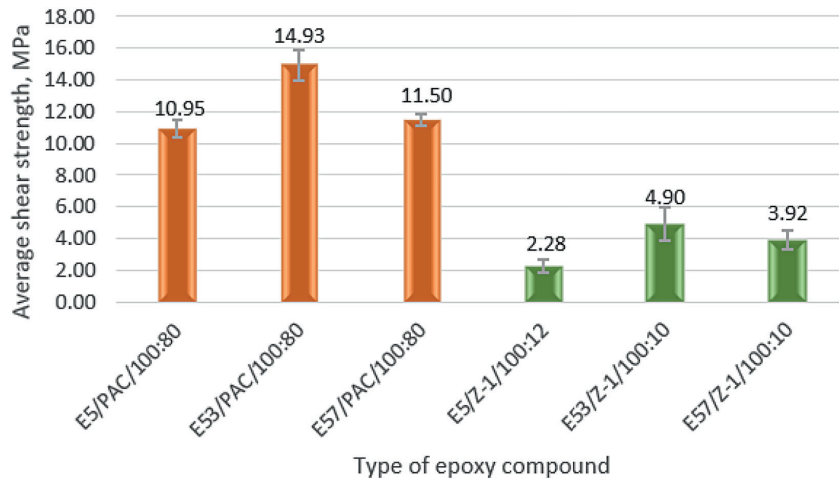


Fig. 2. Average shear strength of the analysed adhesive joints

while the joints made using epoxy adhesive based on unmodified Epidian 5 resin and Z-1 curing agent were characterised by the lowest strength (2.28 MPa). Analysing the strength distribution considering the type of the epoxy compound, in the case of the compound made both with PAC and Z-1 curing agents, the highest strength was found in the case of joints made with Epidian 53 epoxy resin, and the lowest in the case of Epidian 5 epoxy resin. According to the manufacturers, compounds cured with the polyamide PAC curing agent produce a more flexible joint, which can also be observed by analysing the elongation shown in Figure 3, which was measured during the strength tests. For a more accurate comparison, Figure 4 also shows representative tensile/shear curves for the different adhesive joint

variants depending on the adhesive composition used in the adhesive joint.

The elasticity of the adhesive layer consequently determines the strength properties of the adhesive joints. It can be concluded that much higher viscosity of the curing agent, compared to Z-1 curing agent, determines the elasticity of the epoxy compound containing PAC curing agent. After the strength tests, an organoleptic assessment of adhesive bond failure was performed according to EN ISO 10365 standard [43] and the information presented in works [44,45]. The failure of an adhesive bond can occur in four cases:

- detachment of the adhesive layer from the material - adhesive failure, which occurs when the adhesion forces are smaller than the cohesion forces (cohesion) and the external load forces.

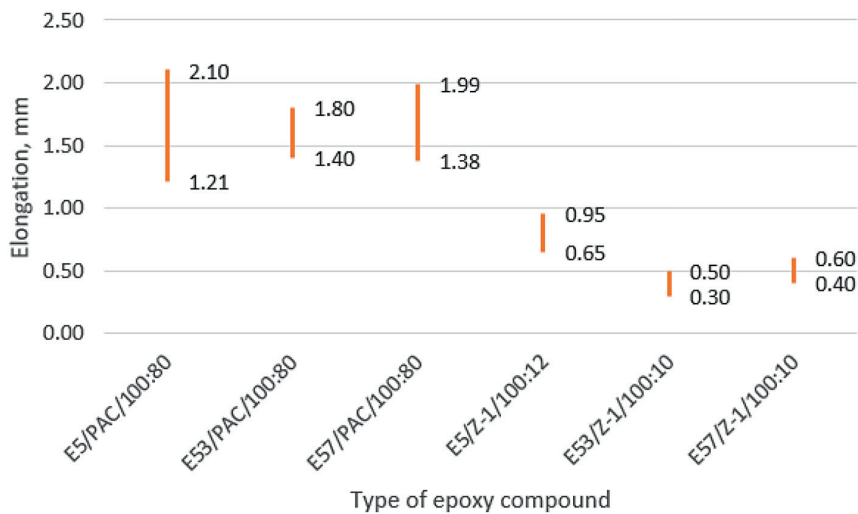
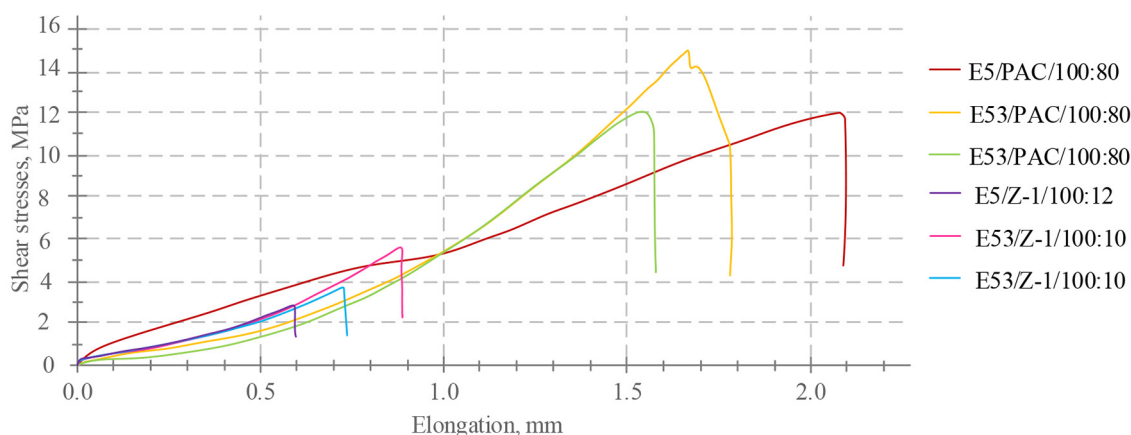


Fig. 3. Elongation measured during tensile testing of adhesive joints



**Fig. 4.** Representative tensile/shear curves for different adhesive joint variants depending on the adhesive composition

According to the standard defining the main patterns of adhesive bonding types – ISO/DIS 10365:1992, designated as AF,

- adhesive bond failure – cohesive failure, which occurs when the intermolecular bonding forces of the adhesive are less than the cohesion forces and the external load forces. According to ISO/DIS 10365:1992, the designation CF,
- partial detachment of the adhesive layer from the material with simultaneous destruction of the adhesive bond – adhesion-coupling failure. According to ISO/DIS 10365:1992 the designation ACF(p),
- failure of the bonded material (adherent) – failure of the structural material which occurs when the adhesive cohesion forces and the adhesion forces are comparable or exceed the cohesion forces of the bonded material. According to ISO/DIS 10365:1992 the designation AF+CF.

Based on visual assessment, it was found that in the analysed adhesive joints, in all cases of adherent joints, damage to the adhesive layer occurred, and the damage was of mixed, adhesive-cohesion nature classified as ACF(p) failure.

For a comprehensive evaluation of the results obtained were subjected to statistical analysis, the results of which are presented in the table below. However, the assumption of the normality of the distribution was checked before selecting the appropriate statistical test. The Shapiro-Wilk test was carried out at a confidence level of  $\alpha = 0.05$ . The test results and the assessment of the normality of the distribution are also summarized in Table 7. The Shapiro-Wilk test showed that the assumption of the normality of the distribution was not met, and therefore the test of non-parametric statistics was used in the following analysis. The Tukey test of significant differences was applied. The results of the test are summarized in Table 8. For a more accurate comparative analysis, Tukey’s homogeneous groups test was additionally performed. The result of this test is presented in Table 9.

The statistical analyses carried out show that the significant differences in the mean values of the shear strength for the individual adhesive joints occurred in the case of: for the epoxy compounds containing the PAC curing agent, between the epoxy compounds based on Epidian 5 epoxy resin, Epidian 57 epoxy resin, and Epidian 53

**Table 7.** Results of Shapiro-Wilk normality test

Epoxy adhesive	Shapiro-Wilk statistics W	Probability level p	Normal distribution
E5/PAC/100:80	0.90	0.44	Yes
E53/PAC/100:80	<b>0.79</b>	<b>0.05</b>	<b>No</b>
E57/PAC/100:80	0.84	0.19	Yes
E5/Z-1/100:12	0.93	0.57	Yes
E53/Z-1/100:10	0.89	0.37	Yes
E57/Z-1/100:10	0.85	0.21	Yes

**Table 8.** Results of Tukey’s test of significant differences

Tukey’s HSD test; Approximate probabilities for post hoc tests Error: Between-group MS = .54176, df = 22.000						
Epoxy adhesive	E5/PAC/100:80	E53/PAC/100:80	E57/PAC/100:80	E5/Z-1/100:12	E53/Z-1/100:10	E57/Z-1/100:10
E5/PAC/100:80		0.000144	<b>0.894818</b>	0.000144	0.000144	0.000144
E53/PAC/100:80	0.000144		0.000146	0.000144	0.000144	0.000144
E57/PAC/100:80	<b>0.894818</b>	0.000146		0.000144	0.000144	0.000144
E5/Z-1/100:12	0.000144	0.000144	0.000144		0.000438	0.032322
E53/Z-1/100:10	0.000144	0.000144	0.000144	0.000438		<b>0.318715</b>
E57/Z-1/100:10	0.000144	0.000144	0.000144	0.032322	<b>0.318715</b>	

**Table 9.** Tukey homogeneous groups test results

Tukey’s HSD test; Homogeneous groups, alpha = .05000 Error: Between-group MS = .54176, df = 22.000				
Adhesive used in the study	Homogeneous groups			
	1	2	3	4
E5/PAC/100:80		****		
E53/PAC/100:80				****
E57/PAC/100:80		****		
E5/Z-1/100:12			****	
E53/Z-1/100:10	****			
E57/Z-1/100:10	****			

epoxy resin. For the adhesive joints made with the epoxy compounds cured with the Z-1 curing agent, the significant differences were observed for adhesive joints made with epoxy resin compounds based on Epidian 5 epoxy resin, and Epidian 53 and Epidian 57 epoxy resins.

**DISCUSSION**

Among the basic technological factors having a significant impact on the adhesive joints strength is: the method of the surfaces treatment of the adherends or the type of the applied adhesive. The selection of an adhesive should be based on the type of application or the type of the joined material. One of the technological indicators determining the properties of the adhesive is its viscosity. The adhesive with a high viscosity is difficult to apply to the surface of bonded materials, it penetrates the material poorly and the applied coating is uneven. Adhesives with too low viscosity at a single application on porous materials give an insufficient film thickness, and in this case, it is necessary to lubricate it several times, which increases its consumption. The optimum adhesive viscosity is adapted to the type of the adherends

and their purpose. Another issue determining the success and high strength of the adhesive joints is appropriate treatment of the joined surface, which in turn also determines the appropriate adhesion of the adhesive to the substrate. Doluk et al. [46] presented that the application of mechanical surface treatment positively influences the strength results obtained. Rudawska et al. and Miturska et al. [47, 48] presented similar conclusions in their paper.

Comparison of the obtained shear strength results show that higher strengths were obtained for the adhesive joints prepared with the epoxy compounds consisting of PAC curing agent, which is characterized by much higher viscosity in comparison with the epoxy compounds consisted Z-1 curing agent. Moreover, it was observed that the highest strengths were obtained in the case of the epoxy compounds consisting of Epidian 53 epoxy resin, which is characterized by the lowest viscosity, thanks to which the consistency of the epoxy adhesive allows it to penetrate all pores and irregularities on the surface intended for bonding. The lowest strengths, considering the type of the resin used, were obtained with Epidian 5 epoxy resin. This is the resin with the highest viscosity, which can be a hindrance during penetration of the adhesive into the irregularities of the surface.



## CONCLUSIONS

During the design and the construction of the adhesive joints, a very important stage is the proper selection of the adhesive, as it significantly affects the strength properties of the adhesive joints. Also important is accuracy of carrying out the activities during the bonding process, both a properly prepared surface for bonding and the correct amount of the adhesive compound components and its uniform distribution on the bonded surface. This work was aimed at determining the influence of the viscosity of the components of adhesive compounds on the strength properties of adhesive joints of 1.0503 steel. The adhesive compounds consisting of three types of the epoxy resins were analysed: Epidian 5, Epidian 53 and Epidian 57, as well as two curing agents of different viscosities: the polyamide (PAC) curing agent and the amine (Z-1) curing agent. Tests were carried out on single-lap sheet adhesive joints, the surfaces of which were subjected to the mechanical treatment with an abrasive coated tool and then degreased with technical acetone.

Based on the experimental studies carried out and the analysis of the results obtained, it was observed that the adhesive joints prepared with the epoxy adhesive compounds containing the PAC curing agent, characterized by 500 times higher viscosity than the Z-1 curing agent, showed significantly higher shear strengths than those joints prepared with the epoxy adhesive compounds containing in their compound the Z-1 curing agent. The adhesive joints made with the epoxy adhesives containing both PAC and Z-1 curing agents with Epidian 53 epoxy resin (with the lowest viscosity among the resins used) were characterized by higher strength in comparison to the adhesive joints made with compounds containing Epidian 5 and Epidian 57 epoxy resin. The use of PAC curing agent in the adhesive compounds with the epoxy resins results in a more elastic joint than the use of Z-1 curing agent.

The presented information may have a significant impact on planning the joining technology of components made of steel sheet of the grade 1.0503. In the future, research is planned on the preparation of the adhesive compounds to order to obtain a favourable adhesive viscosity, increasing the adhesive contact with the joined surface.

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