Determination of design mechanical properties of adhesives in a tensile and compression test

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Abstract. Structural adhesives are increasingly used in the construction of mechanical devices. Adhesive joints replace welded, soldered and other joints. The strength of the adhesive joints obtained allows the transfer of loads. The possibility of combining different materials is a significant advantage. In order to design joints, it is necessary to know the mechanical properties of adhesives. Determining them requires the use of special methods of obtaining specimens. Analytical and numerical methods of stress estimation in adhesive joints require more material data than is provided by manufacturers. The aim of the following work is to present the method of manufacturing specimens and to compare methods for determining mechanical properties based on three example structural adhesives. The paper presents two methods for determining the mechanical properties of exemplary structural adhesives: metacryl, epoxy and polyurethane adhesive. The tests were carried out on a cast and then machined specimens. Flat specimens were used in a tensile test. In compression test, round specimens were used. The results obtained from both tests were compared with the manufacturer's data. Differences and possible reasons for them were indicated. Stress distributions in the adhesive single lap joint were estimated using the Volkersen analytical method for various determined mechanical properties.

1. Introduction

Adhesive joints replace welded, soldered and other joints. The strength of the adhesive joints obtained allows the transfer of loads. The possibility of combining different materials is a significant advantage [1-2].

This makes these connections more and more widely used in aviation, the automotive industry and shipbuilding. Structural joints must have a certain strength and fatigue durability. In computational methods, it is assumed that the stresses occurring in the material must be less than or equal to the acceptable stress levels. The stress distributions occurring in the joints is not uniform over their entire surface. The unevenness of the distribution is influenced by the asymmetry of the connection, the rigidity of the joined elements, the mechanical properties of the adhesive, the nature of the load and others factors. There are analytical methods for determining stress distributions in joints, and increasingly used, numerical methods. All methods require the definition of mechanical properties of adhesives such as Young's modulus, Poisson's ratio or Kirchhoff's module. In numerical methods, it is possible to implement the entire stress plot as a function of strain [3-5].

Determination of mechanical properties of adhesives is associated with several technological problems. The first one is to obtain specimens of high adhesion material. Therefore, the molds must be

made of materials difficult to glue, eg. Teflon, polyethene, silicone [6]. Adhesive materials differ in viscosity in the open state and shrinkage in the cross-linking process, which means that it is not possible to use one universal method for obtaining repeatable specimens. The second problem is to obtain a specimens with properties similar to those of the adhesive layer in joints. In particular, the applied pressure, the amount of heat generated by the exothermic cross-linking process and the possibility of giving it to the combined elements may be of importance here [7-8].

The aim of the article is to present the method of manufacturing specimens and to compare methods for determining mechanical properties based on three example structural adhesives. The paper presents two methods for determining the mechanical properties of exemplary structural adhesives: metacryl, epoxy and polyurethane adhesive in a tensile and compression test. The results obtained from both tests were compared with the manufacturer's data.

2. Methods

2.1. Material and method of specimens preparation

The material for testing was two-component structural adhesives for joining metals, plastics and ceramics: Plexus MA300 methacrylic adhesive, Epidian 53 epoxy adhesive cured with Z1 hardener and Multibond 3111 polyurethane adhesive. The specimens for determining static mechanical properties in a tension test were designed in accordance with the PN-EN ISO 527-2-1998 standard, using the dimensioning for small 1BB shape. The dimensions of the specimens are shown in the Figure 1a. The specimens for determining static mechanical properties in a compression test were designed in accordance with the PN-EN ISO 604-2006 standard. The dimensions of the specimens are shown in the Figure 1b.
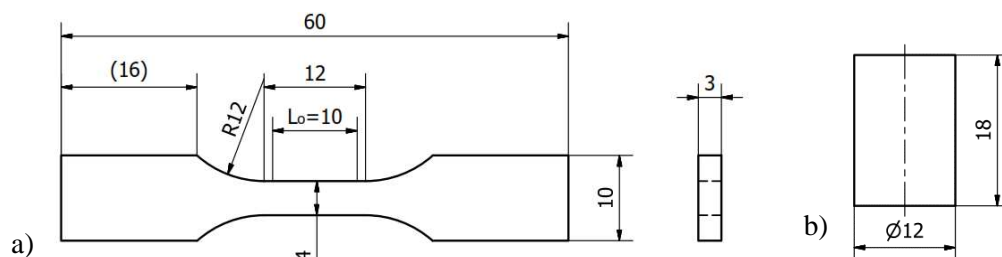

Figure 1. Dimensions of the test specimens for mechanical properties: a) under static tension in accordance with PN-EN ISO 527-2-1998; b) under static compression in accordance with PN-EN ISO 604-2006.

Specimens of methacrylic adhesive and epoxy adhesive were made of cast plates with dimensions of 75 mm x 45 mm x 5 mm in the form. The paste methacrylic adhesive was applied closer to one edge of the mold, and then the mold was closed at a slight angle to distribute the adhesive inside, as shown in Figure 2a. This method of application reduces the risk of air bubbles in the casting. To avoid sticking the molded material to the mold, its interior consisted of Teflon plate and a silicone frame. Epidian 53 epoxy adhesive is characterized by low viscosity and a long time to obtain initial strength. Due to the fact, that its consistency is liquid, the venting of the casting takes place spontaneously under the influence of gravity. It was cast in an open mold shown in Figure 2b. After curing, cast plates of methacrylic and epoxy adhesive were removed from the molds. Then, the target shape of the samples was milled from the plates.

After hardening, polyurethane adhesives are characterized by high flexibility, which makes milling impossible. Therefore, specimens of polyurethane adhesive were cast in molds in the shape and dimensions of the final specimens shown in Figure 2c. Polyurethane adhesive is characterized by medium viscosity and medium time to obtain initial strength. The form consisted of Teflon with milled outlines of specimens, two silicone spacers placed bellow and above Teflon and two plates between which the whole was clamped together. The adhesive was applied to the gripping parts of the

specimens. On the one side, a larger amount was applied, which resulted in filling the measuring part of the specimen during the closing of the mold. This method of obtaining minimized the number of defects. The specimens for testing the mechanical properties in compression were cylinders whose diameter was 12 mm. They were manufactured in the polyethylene forms shown in the Figure 2d. Specimens shown in Figure 3, were seasoned for one month in room conditions (20 ± 2ºC, humidity $45 \pm 5\%$) and then tested.
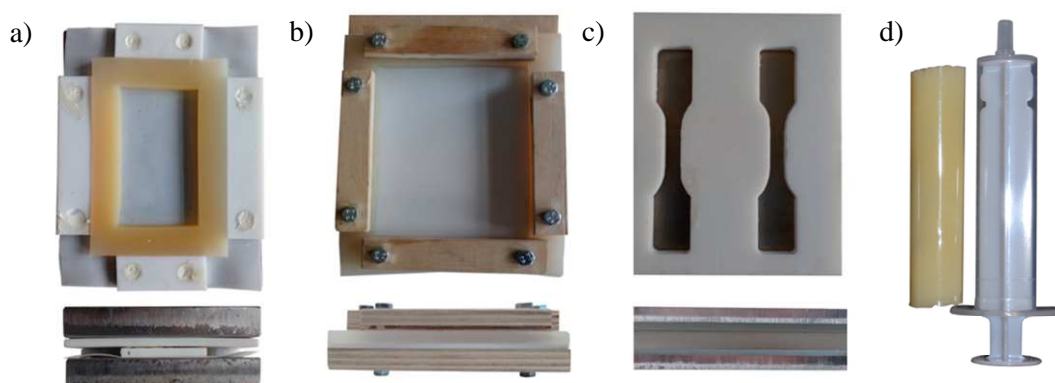

Figure 2. Molds for casting adhesives: a) methacrylic Plexus MA300; b) epoxy Epidian 53; c) polyurethane Multibond 3111; d) cylinders for testing mechanical properties in compression.
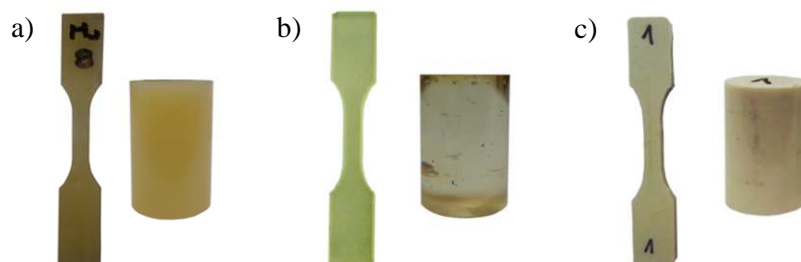

Figure 3. Specimens of: a) methacrylic adhesive Plexus MA300; b) epoxy adhesive Epidian 53+Z1; c) polyurethane Multibond 3111.

2.2. Test method

The specimens in tensile test, were fixed in the mechanical jaw holders of the INSTRON 5966 strength testing machine shown in Figure 4a, with a force measuring device with a range of up to 10 kN. During the tests the displacement parameter for a constant value was accepted in both directions to be equal to 0.005 mms^{-1}. Two extensometers were used during the tests. The first extensometer for
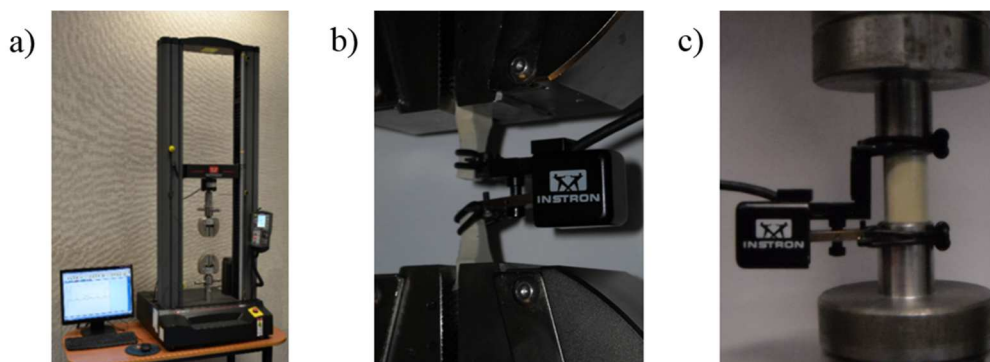

Figure 4. a) INSTRON 5966 testing machine; b) tensile test and speciemns in holders; c) compression test and specimens between the pressure devices.

measuring longitudinal strain with 10 mm measurement base and a measuring range of ± 1 mm as shown in the Figure 4b. The second extensometer for measuring transverse deformations with an adjustable measuring base and measuring range of ± 0.5 mm. In the compression test, specimens were fixed between the pressure devices as shown in the Figure 4c. The extensometer for measuring longitudinal strain with 25 mm measurement base and a measuring range of ± 5 mm was used during the test. The tests were carried out on 5 specimens of each material in ambient conditions.

2.3. Test results
The graphs (Figure 5) present examples of stress as a function of longitudinal strain for the tested materials. In the tensile test, each of the specimens was destroyed. In the compression test, only specimens of epoxy adhesive were destroyed. Specimens of methacrylic and polyurethane adhesives sustain significant transverse deformations, which makes the continuation of the test unnecessary. The determined Young's modulus from tensile tests and compression tests for the materials assumed similar values. For the methacrylic and epoxy adhesives, the yield point can be determined in the form of stress values at which the deformation occurs without further load increase. For the polyurethane glue, the yield stress was determined at 0.2% permanent strain. In the compression test, higher yield strength values were obtained than in the tensile test. Table 1 presents the average results of tests of mechanical properties of structural methacrylic adhesive Plexus MA300, epoxy Epidian 53 and polyurethane Multibond 3111. The data specified by the manufacturers are given in brackets.
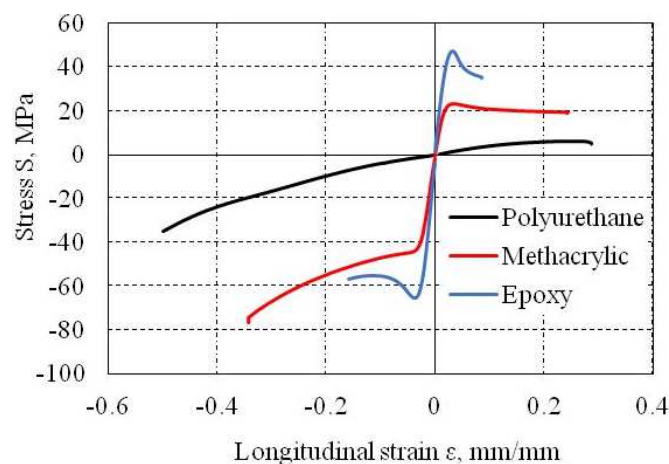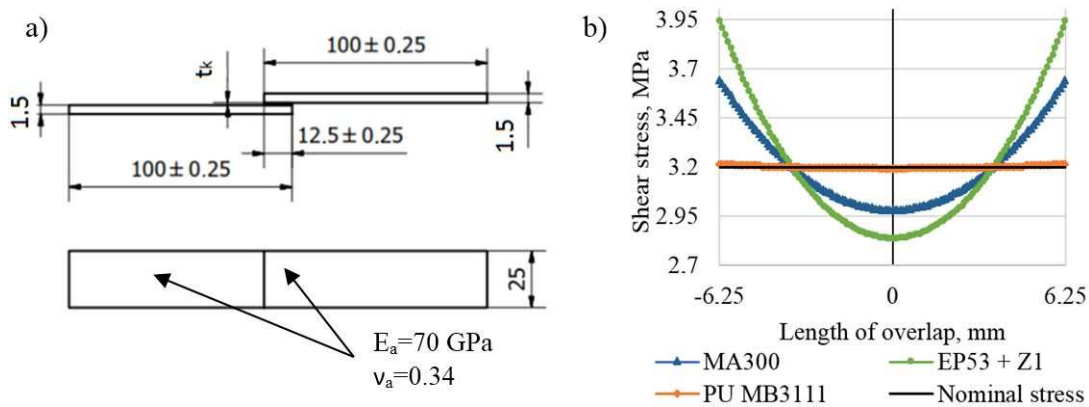

Figure 5. Stress diagram as a function of strain of Plexus MA300 methacrylic adhesive; Epidian 53 + Z1 epoxy adhesive and Multibond 3111 polyurethane adhesive.

In the overlap joints, the distribution of shear stresses in the sample is not constant along the entire length of the overlap. The difference between the average stress calculated from the formula and the local stress accumulation at the ends of the overlap depends on many factors, including the thickness of the adhesive layer and its Kirchhoff module G_k. The dependence allowing to determine stress distribution was given by Volkersen [9-10]. Figure 6a shows the dimensions of the single-lap joint in accordance with the standard PN-EN 1465:2009 for determining the mechanical properties of adhesive under shear. The stress distributions for the same exemplary loading force of 1000 N were calculated.

The tested adhesives have different Kirchhoff's value which results in significant differences in stress values at the ends of the overlap (Figure 6b). Adhesives with lower stiffness allow for a more even value of shear stresses in the overlap joint. Uneven stresses in the joint can be described by the parameter β, which is the ratio of the shear tensions in the joint to the nominal values. The influence of the adhesives thickness t_k and Kirchhoff module values on stress inequality values is presented in the graph (Figure 7a). The parameter β is larger for joints with a thin layer of adhesives and adhesives with higher stiffness.

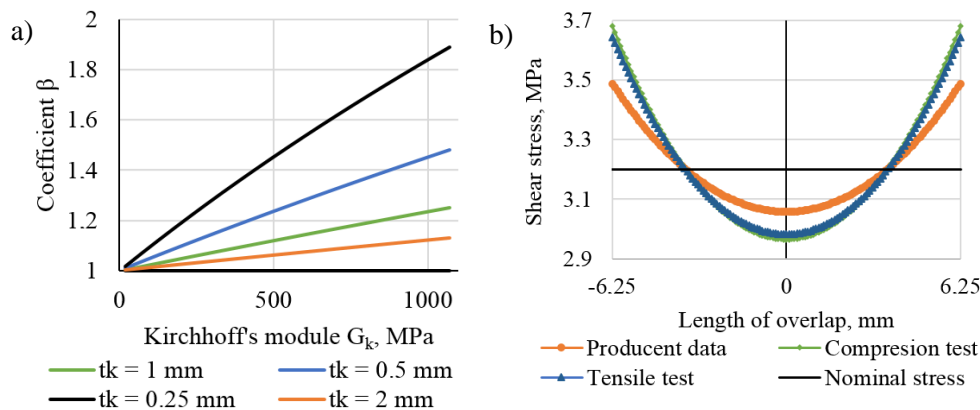**Table 1.** Determined mechanical properties of the tested adhesives.

Material properties	Methacrylic Plexus MA300		Epoxy Epidian 53+Z1		Polyurethane Multibond 3111	
	Tensile	Compression	Tensile	Compression	Tensile	Compression
Tensile strength (MPa)	23.7 (22)	45.8 -	47.1 (50)	73.2 -	6.56 -	45.8 -
Yield strength (MPa)	23.7	-	47.1	-	$2.58^{0.2}$	-
Young's modulus E_k (MPa)	1610.5 (1034)	1748.2 -	2730.6 (2500)	2647.4 -	51.4 100	39.8 -
Poisson ratio v_k (-)	0.4	-	0.39	-	0.45	-

Figure 6. a) Dimension of single-lap specimen; b) shear stress distribution along overlap of single-lap adhesive joint.

The material data determined in the tensile test, compression and the material data provided by the manufacturer may introduce an error in the determination of the values of local stresses at the ends of the overlap. The error resulting from the use of the above material data was estimated. The values of the determined stress uneven parameters for all adhesives, for a standard overlap joint with an adhesive thickness of 1 mm are shown in Table 2. The differences ΔE_k expressed in %, in the determined Young's modules were calculated relative to the values given by the manufactures. They are the largest for methacrylic and polyurethane adhesives and amount to as much as 70%. The differences between the test results of tensile and compression are much smaller and amount to a maximum of 14%. Such variations in the obtained results affect to a lesser extent the value of variability in the distribution of stresses in the considered joint. The maximum difference was calculated for the methacrylic adhesive. Taking into account the manufacturer's data and the data determined in the own research, the difference was 4.5% for the tensile test and 5.5% for the compression test. Similar differences in the determined Young's Module for the more susceptible polyurethane adhesives gave differences in the determined maximum shear stresses at the respective 0.2% and 0.1%. For MA300 methacrylate adhesive differences in stress distributions for various material data are shown in the graph (Figure 7b).

Table 2. Determined mechanical properties of the tested adhesives.

	Young's module E_k (MPa)	ΔE_k (%)	Poisson ratio v_k (-)	Kirchhoff's module G_k (MPa)	Max shear stress T_{max}	B (-)	ΔT_{max} (%)
	Metacrylic						
P[a]	1034.0	-	0.40	369.3	3.488	1.090	-
T[b]	1610.5	55.8	0.40	575.2	3.644	1.139	4.5
C[c]	1748.2	69.1	0.40	624.4	3.681	1.150	5.5
	Epoxy						
P[a]	2500.0	-	0.39	899.3	3.884	1.214	-
T[b]	2730.6	9.2	0.39	982.2	3.944	1.233	1.6
C[c]	2647.4	5.9	0.39	952.3	3.922	1.226	1.0
	Polyurethane						
P[a]	100.0	-	0.45	10.0	3.208	1.002	-
T[b]	51.4	-48.6	0.45	17.7	3.214	1.004	0.2
C[c]	39.8	-60.2	0.45	13.7	3.211	1.003	0.1

[a] Manufacture's data.
[b] Data from tensile test.
[c] Data from compression test.

Figure 7. a) Value of the coefficient β as a function of the Kirchhoff module; b) distribution of shear stresses in the overlap joint for different material data values.

3. Conclusion

The method of manufacturing specimens used in the presented studies obtaining obtain reproducible results of mechanical properties of tested structural adhesives significantly differing in their viscosity. Determination of Young's modulus for adhesives in a tensile and compression test gives similar results. The determined limits of strength and yield, as well as maximum deformations, are greater in the compression test than in the tensile test. Thus, it is not possible to use both methods interchangeably to determine these three mechanical properties. The highest tensile strength was achieved by epoxy and methacrylic adhesives. A more even distribution of stresses in the bonded joint can be achieved by using adhesives with less stiffness or by increasing the thickness of the adhesive layer. The error value in the determined Young's modulus causes a significantly smaller error in the calculated value of the maximum tangential stresses in the overlap joints. This error decreases with the adhesive susceptibility.

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