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Experimental Testing of the Effect of PEEK Surface Treatments on the Shear Strength of Glued Joints

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Abstract. Adhesive bonding is a way of joining two components together. Bonded joints have many advantages in comparison with other types of joints (bolts, rivets, welds, etc.), such as low weight, more uniform stress distributions, easy manufacture and the possibility to join dissimilar materials, possibility to join very thin adherents and very large surfaces. On the other hand, bonded joints also have a number of disadvantages. They are not suitable for structures subjected to fatigue, require surface preparation and have limited stability to elevated temperatures. With the rapid development of the properties of adhesives (higher peel and shear strength and also allowable ductility up to failure), their use in all areas of industry has expanded.

Introduction and motivation

One of the areas of research activities in the Design and Development Department of Space Division in the Czech Aerospace Research Centre is 3D printing focused on Polyetheretherketon (PEEK). PEEK is a semi-crystalline organic thermoplastic with glass transition temperature of approximately 143 °C and melting temperature of around 343 °C. Due to its properties, PEEK is used in aerospace, chemical and pharmaceutical industry and is expected to be used in more important areas. PEEK is also used for ultra-high vacuum applications thanks to very low outgassing. Most plastics contain volatile organic compounds which, when vaporized, are poisonous chemicals. Direct contact with these compounds can cause serious health problems. High temperatures usually accelerate outgassing. Since many industrial applications involve high temperatures, plastic outgassing becomes a critical factor in choosing plastic materials. PEEK is also used for good mechanical properties. The yield strength of PEEK is approximately 110 MPa, Young modulus is 4400 MPa and specific mass is 1.32 g/m^3 [1, 2].

PEEK is also suitable for 3D printing. However, performance of most of the 3D printers is not sufficient to melt PEEK. In comparison to other materials widely used in 3D printing, PEEK has a higher melting point (343 °C). To solve this problem, it is necessary to apply a nozzle heated to about 400 °C [2]. In the Design and Development Department of Space Division in the Czech Aerospace Research Centre, 3D printing using PEEK based on the FFF technology is used for the development of spacecraft elements. A typical example is presented in Fig. 1. To connect this honeycomb with skins based on thermoset matrix composite, it is necessary to use some adhesive. Using of adhesives is necessary in more applications where

PEEK parts are bonded together or to other materials. This is the main motivation for the development of surface treatments in order to improve the properties of bonded joints of PEEK 3D printed parts. Also PEEK parts made by injection are used in the design of aerospace structures, but this technology does not offer so big freedom in the choice of the shape.

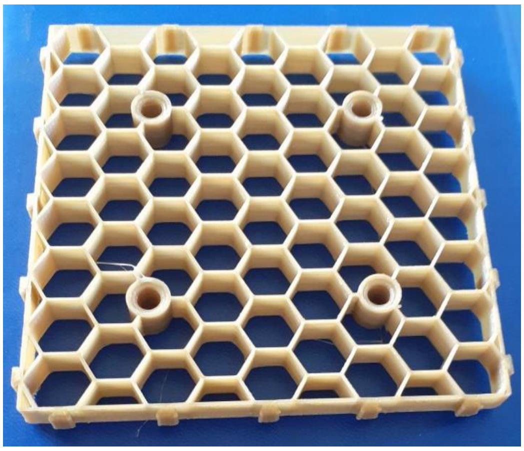

Fig. 1: 3D printed honeycomb for sandwich panel made of PEEK with integrated places for plastic inserts

As already mentioned structural bonding is frequently used today for connecting two components together. This connection must ensure a satisfactory structural strength and good durability. A good design of bonded connection is a necessary condition for reliable function. Design of bonded connections with many useful information are described in [3].

It is generally known that bonding of plastics can cause problems especially in the case of plastics with low surface energy [4]. Today adhesives for these types of plastics are available. But there are not many information in terms of bonding of PEEK with PEEK or with other plastics. Mechanical properties of these connections of PEEK are not described on a satisfactory level.

Description of test specimens, used adhesive and methods

Test specimens were not designed in the line with recommendations of commonly used standards. This approach is possible thanks to the fact, that only comparison of individual surface treatments was expected. Tests were not performed in order to obtain design allowables, where the conformity with standards is necessary.

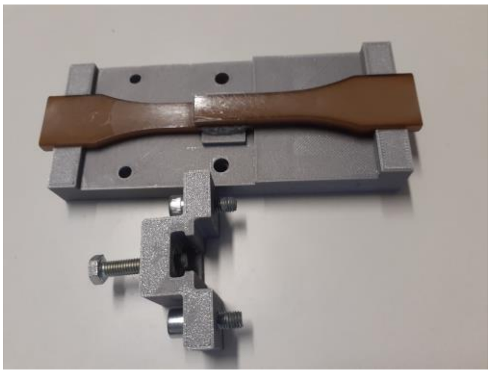

Fig. 2: Test specimen in the jig after gluing (left figure) and final test specimens before testing (right figure)

Test specimens have a typical “dog bone” shape. To ensure a good repeatability of test specimens during the process of gluing a special jig was designed and manufactured using 3D printing, see Fig. 2. Trial tests started with specimens made by injection. The motivation for this was an effort to compare results with other results available in the literature. Bonding of PEEK 3D printed parts is discussed in the literary sources only minimally.

During trial tests, three surface treatments in combination with one type of adhesive were tested. 3M™ Scotch-Weld™ Structural Plastic Adhesive DP8005 was used for the preparation of test specimens. It is a two-part acrylic adhesive specially formulated to bond low surface energy plastics without priming. Mechanical properties can be found in [5]. Based on manufacturer information, see [5], typical strength reached in single lap shear test is 16.3 MPa for joint of two FRP adherends, where cohesion failure was reached. Other example is a joint of two ABS adherends where the strength is 6.7 MPa and adhesion failure was reached.

In terms of surface treatments, three types including acetone degreasing (immersion in a bath for 5 minutes, acetone, puriss. p.a.), sandblasting with acetone degreasing (blasting agent Alu. oxide 120 mesh, pressure 2 bar, distance 0.3 m, blasting angle 45 ° and then immersion in a bath for 5 minutes, acetone, puriss. p.a.) and nickel plating (activating bath 20% H_2SO_4 was used for 5 minutes prior to chemical Ni plating – 20 minutes in 10% $NiSO_4 \cdot 6H_2O$ with 5% NaH_2PO_2 water solution at 50 °C) were tested till now.

Test specimens were subjected to a single lap-shear test according to the standard ASTM D3163-01(2014) [6]. Test set-up is shown in Fig. 3. Test speed was 1,3 mm/s. Since only baseline screening tests were performed, three test specimens of each type of surface treatment were tested with some exceptions.

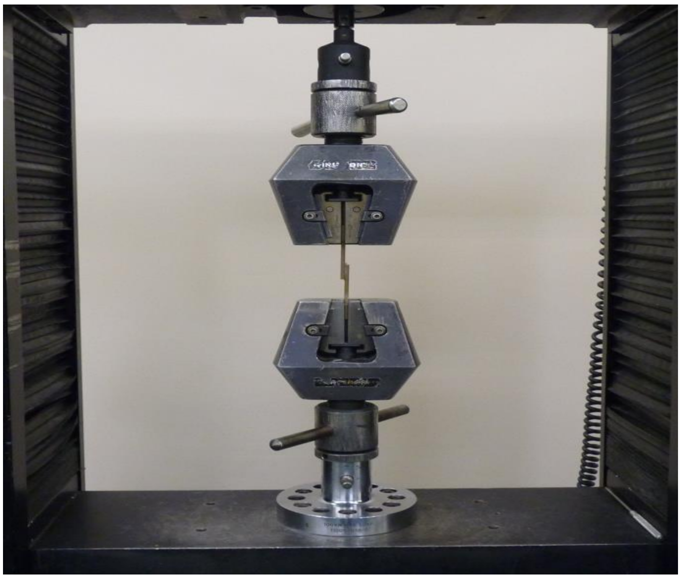

Fig. 3: Single lap-shear test - the test set-up

Discussion of the results

The results are presented in Table 1. The evaluation was performed on the basis of ASTM D3163-01. The original idea was to test three test specimens for each surface treatment type during trial tests. This plan was followed only in the case of sandblasting, see Tab. 1. The results for nickel plating are determined only on the basis of two specimens and therefore have only informative character. More tests will be performed to obtain a satisfactory statistical sample. It can be seen that the coefficient of variation is very low. This is probably caused by the good repeatability of test specimens thanks to the jig for their preparation and also good repeatability of the surface treatments quality. The highest apparent shear strength was reached in the case of sandblasting surface treatment. This finding is in the line with generally known fact, that sandblasting has a positive effect on the shear strength of glued joints.

Good results were also obtained in the case of nickel plating surface treatment although the apparent shear strength is significantly lower. Acetone degreasing without any other treatment shows the lowest strength and the highest scatter in results, which was expected.

Test specimens after testing are presented in Fig. 4. Failure mode of the specimens can be defined as cohesive failure of the adhesive. In some cases the combination of cohesive failure of adhesive and the adhesion failure of adhesive was determined. Cohesive failure of adhesive is a valid failure mode. More information about valid failure modes during testing of shear strength of glued joints can be fined e.g. in [7].

Tab. 1: Results of apparent shear strength testing with statistical evaluation

	Sandblasting and Acetone degreasing [MPa]	Acetone degreasing [MPa]	Nickel plating [MPa]
1	6.37	3.89	4.92
2	6.86	3.70	4.80
3	6.93	4.03	-
4	-	3.31	-
Mean [MPa]	6.72	3.73	4.86
Standard dev. [MPa]	0.305	0.313	0.085
Coef. of variation [%]	4.54	8.37	1.75

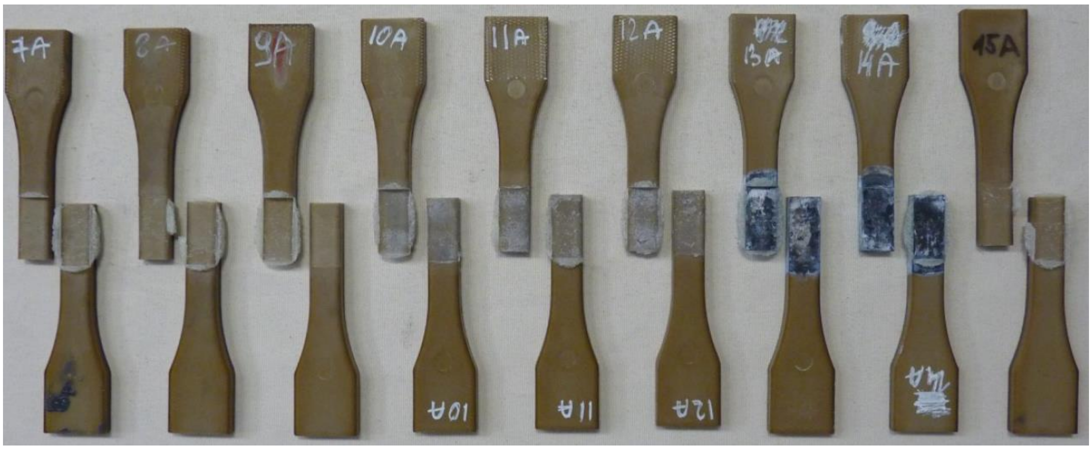

Fig. 4: Test specimens after the test

Conclusions

Presented results show that it is possible to realize the glued joint between PEEK adherends using common adhesive. Appropriate surface treatment is a necessary step before gluing. Although the statistical sample was low in these baseline experiments, the results show, that it is possible to affect the properties of PEEK surface before gluing and get a satisfactory results.

The results show a very low scatter in apparent strength values. In terms of experimental determination of design allowables it is a positive finding. For the calculation of design allowables, both mean value and standard deviation are necessary. With increase in standard deviation, decrease in design allowables is obtained. Generally, low scatter during measuring of mechanical properties is a good expectation for the design of reliable structure. Single lap shear test is used for the comparison of the effect of surface treatments. In order to get more precise strength values, double lap shear will be performed.

In other step of the research, another surface treatment using plasma will be tested. It is generally known fact that plasma surface treatment has a positive effect on the surface properties in terms of glued joints.

In terms of tested adhesives it is expected to test some foil adhesive. Foil adhesives are used for the manufacturing of sandwich panels. The use of these panels in the design of aircraft and spacecraft structures is very frequent.

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