Analysis of the effect of selected factors on the strength of adhesive joints

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Abstract. Adhesive joining is one of the well-established methods for joining structural materials, however it was not until the rapid development in chemistry of adhesives that it could gain wide recognition. It is impossible to imagine any branch of modern industry without adhesive joining technology, which proves indispensable in: aircraft, aerospace, automotive or building industries. The importance of adhesive joining and adhesive materials in the economy of modern world is unparalleled. Among the factors of crucial importance to the strength of adhesive joining it is the preparation of adhered surfaces through proper treatment that appears to predominate. The issue is even more important as the decision regarding the surface treatment is undertaken by a technologist, whereas the other elements of joining technology, such as temperature, time or load applied during cure, and the joint seasoning time are known and specified by manufacturers for each adhesive. Another factor relevant to the strength of adhesive joints is the effect of cyclic thermal loading. The disparity in the coefficient of thermal expansion of adhesives and adherends and in their thermal conductivity results in the adhesive joint being subjected to cyclic internal stresses. Thermal fatigue of cured adhesives may implicate ageing processes, increase polydispersity of the substrate and consequently affect its mechanical properties. This paper presents the results of comparative analysis of the shear strength of adhesive lap joints. The tests were performed on samples following different surface treatment methods, which were subsequently subjected to thermal shock in the varying range of temperatures between -40 °C and +60 °C.

1. Introduction

Adhesive joints are frequently applied to bond and seal mechanical structures. This joint type is preferable due to the fact that adhesives offer numerous advantages over other joining solutions. Performance of adhesive joints depends on several factors, among which this is the type of surface treatment that plays a vital role in the preparation of surface of adherends [1-3]. By definition, surface treatment technologies produce desired surface texture, i.e. optimised in geometrical sense, of a non-oriented structure, and establishing appropriate energy conditions of the surface layer, by which we mean a uniform energy state on the entire surface of the adhesive joint area [2-4].

Analysis of selected characteristics of adherends surface geometry, and surface roughness parameters in particular, is significant [5, 6] especially in terms of preparing the surface layer for the technology utilizing adhesion. The growing use of adhesive joining is attributed to the advantages over other methods that the technology provides. Adhesive joining primarily optimises costs of assembly or production of technical objects, owing to the low cost of producing adhesive joints. Another advantage is that adhesion technology is capable of joining homogenous materials of different properties, sizes,
shapes and dimensions. A wide variety of commercially available adhesives differing in the chemical composition enables selecting an optimal adhesive for a particular adherend.

Unlike other joining methods, such as welding or pressure welding, adhesive joining does not affect the structure of adherends, nor does it require high temperature in the bond area, which could induce thermal deformation. In relation to riveted joints, adhesive technology does not require drilling holes in the joint area, which may have an adverse effect on the strength of material and generate unfavourable stress concentration in the joint [7]. Forming joints, adhesives absorb vibrations, seal and air- or water-tighten the joint.

The growing attention to adhesive bonding in joints subject to thermomechanical loading results from the continuous progress in the adhesive engineering and the knowledge capital regarding strength of adhesives. Thermal fatigue of adhesive joints and its effect on their long-term performance and safety is under ongoing analysis; however, the results of studies in the field may not be fully reliable, due to the sheer number of factors affecting the adhesive and cohesive strength of joints. The disparity in the coefficient of thermal expansion, or in the thermal conductivity coefficient of the adhesive and adherends, lead to the occurrence of cyclic loading induced by internal stresses. Thermal fatigue of cured adhesive material may imply ageing processes, which are difficult to estimate, particularly in critical applications, such as in the aerostructures.

Recent developments in 3D printing have led to manufacturing ready-made elements, which are often designed for the purpose of adhesive joining, despite the small workspace of 3D printers [8]. This type of elements is becoming increasingly popular in a number of applications, including structural elements.

2. Test methodology
Table 1 collates variants of surface treatments applied to 316L steel substrate. There were 5 variants of 100x25 mm and 1.5 mm thick samples that were subjected to tests. In order to generate desired surface texture and to remove the physisorption layer, a part of the samples was mechanically treated by means of sand-blasting, conducted at the pressure of 0.8 MPa. The samples treatment process was twofold: initially the samples were rinsed in a degreasing agent and dried with a paper towel, both repeated twice; secondly, the samples were cleaned with Loctite 7061 and left to dry. Table 2 shows the chemical composition of 316L steel substrate, based on the material certificate. Table 3 shows selected properties of 316L steel, based on the material certificate.

| Variant | Surface treatment          |
|---------|---------------------------|
| T1      | Untreated                 |
| T2      | Abrasive granular tool P320|
| T3      | Abrasive cloth P280       |
| T4      | Abrasive cloth P100       |
| T5      | Sand-blasting             |

| 316L steel |
|------------|
| Element    | C  | Si  | Mn  | P   | S   | Ni  | Cr  | Mo  | N   |
| Value [%]  | 0,011 | 0,54 | 1,03 | 0,040 | 0,001 | 10,18 | 16,71 | 2,05 | 0,020 |

| 316L steel |
|------------|
| Tensile strength [MPa] | Yield strength [MPa] | Hardness [HV] |
| 592         | 290         | 148          |
Contact angle measurements were taken with a PGX goniometer (Fibro Systems AB, Switzerland) thus providing the base for the estimation of the surface free energy. The measuring liquids were applied on the surface of samples automatically by means of the goniometer, in the form of a 4 µl tensile drop. The two measuring liquids were applied in tests: distilled water (the polar component $\gamma_{pw}=51.0$ [mJ/m$^2$], the dispersive component $\gamma_{dw}=21.8$ [mJ/m$^2$], the surface free energy $\gamma_w=72.8$ [mJ/m$^2$]) and diiodomethane (the polar component $\gamma_{pd}=2.3$ [mJ/m$^2$], the dispersive component $\gamma_{dd}=48.5$ [mJ/m$^2$]). The contact angle measurements were taken at minimum 10 times for each test sample, at ambient temperature of 21-22°C and relative humidity ranged between 40-45%.

The set-up utilised in the surface roughness measurements conducted in the study included roughness, contour and 3D topography measurement system T8000 RC-120-400 by Hommel-Etamic. The system was fitted with a 2 µm contour probe. The roughness sampling cut-off was obtained from literature [9]. The specimens were scanned on the area of 4.8 mm x 4.8 mm. 2D surface roughness parameters were taken in ten repetitions for all surface treatment variants; their mean values are presented in the Table 5. The conducted tests focused on the following 3D area roughness parameters of the surface: $S_q$ – root mean square height, $S_z$ – maximum height, $S_a$ – arithmetical mean height. Moreover, the tests involved the measurement of the following 2D surface roughness parameters: $R_a$ – arithmetic mean deviation of the roughness profile, $R_z$ – maximum height of the profile, $R_t$ – total height of the profile.

The instrument used to produce an image of 316L steel substrate surface was Keyence VHX-5000 microscope.

The tested single-lap adhesive joint samples were formed with the bondline thickness amounting to $g_k= (0.07\pm0.02)$ mm. The remaining dimensions of the joints are show in Figure 1.

![Figure 1. Schematic representation of the tested adhesive joint – single lap specimen.](image)

The test samples were bonded with Hysol 9466 and Hysol 9484 adhesive compositions, produced by Loctite, at ambient temperature range of 21-23°C, and relative humidity between 35-45%. Upon application of adhesive the samples were subjected to loading at the pressure of 0.2 MPa, and left to cure for 120 h. Table 4 shows selected properties of the fully cured adhesives used in tests.

| Physical properties                      | Hysol 9466 | Hysol 9484 |
|----------------------------------------|------------|------------|
| Tensile strength (ASTM D882) [N/mm²]   | 32         | 15         |
| Elongation (ASTM D882) [%]             | 3          | 50         |
| Tensile modulus (ASTM D882) [N/mm²]   | 1718       | 161        |
| Shore hardness (ASTM D1706), Shore D   | 60         | 55         |
The tests were divided into two parts. First, the objective of tests was to determine the effect of surface treatment on adhesive joint strength. According to Table 1, the samples were subjected to one of the five variants of surface treatment (variants T1-T5). The first part of the tests was performed on samples bonded with Hysol 9466. The second part of the tests was devoted to determining the impact of thermal loading (thermal shocks) on the durability of the structure. Single-lap adhesive joints were subjected to thermal shock cycling tests in a thermal shock testing chamber and in the number of 500 and 1000 cycles. At this stage of the tests, we analysed two adhesive compositions of different properties. The range of temperatures applied in thermal loading tests was quite wide, from -40°C to +60°C, hence the temperature gradient was equal to 100°C. The conditioning time for all samples and temperature variants was 15 min, not including the time required for the temperature to stabilise.

Shear strength tests of single-lap adhesive joints were performed on Zwick/Roell Z 150 materials testing machine, in accordance with DIN EN 1465. The crosshead speed in the destructive test was 2 mm/minute with 85 mm distance between holding fixtures in initial position. The sample population was equal to 10.

3. Test results
The first analysed factor was the impact of different surface treatment variants of 316L steel substrate on the adhesive joint strength of samples. Table 5 shows selected 2D surface and 3D area roughness parameters of 316L steel samples measured after the tested surface treatment variants.

Table 5. Selected surface roughness and area roughness parameters.

| Variant | Parameters of surface roughness 2D | Parameters of surface roughness 3D |
|---------|-----------------------------------|-----------------------------------|
|         | Ra      | Rz      | Rt      | Sq      | Sz      | Sa      |
| T1      | 0,07    | 0,90    | 1,38    | 0,376   | 5,60    | 0,287   |
| T2      | 0,19    | 1,99    | 2,89    | 0,292   | 3,10    | 0,228   |
| T3      | 0,14    | 1,45    | 2,20    | 0,214   | 5,48    | 0,158   |
| T4      | 0,16    | 1,42    | 2,43    | 0,270   | 4,87    | 0,212   |
| T5      | 2,10    | 14,09   | 18,25   | 2,20    | 27,2    | 1,73    |

Table 6 shows 3D isomeric images of 316L steel sample surface after different surface treatments. The conducted observations indicate that variant T5 of surface treatment (sand-blasting) produces the most desired surface texture of the tested substrate material.

Figure 2 shows the impact of different treatments applied to the surface of 316L steel substrate material on the strength of single-lap adhesive joints. With a view to limiting the impact of any external factors on readings, the only adhesive composition tested in this part of tests was Hysol 9466, which cured, provides a considerably rigid bondline.

The conducted tests have shown a significant effect of surface treatment technology on the strength of adhesive joints of 316L steel substrates. Compared with untreated samples (T1), a 100% growth in the joint strength was observed for surface treatment variant T2. Furthermore, the mean strength of joints was additionally 10% higher, compared to T2, when the substrate was subjected to surface treatment variant T5. However, the highest increase in the mean strength of adhesive joints was observed in joints treated with T5 relative to T3, where it amounted to 12%.

The second stage of focused on evaluating the effect of cyclic thermal loading on the adhesive joint strength of analysed samples. Bearing in mind the consistency of results it was resolved that the 316L substrate specimens would be subjected to only one surface treatment variant, T2 (Table 1). The impact of thermal shock on joint strength was analysed for two significantly dissimilar adhesive compositions (Table 4).
Table 6. Isomeric and photographic images of 316L substrate surface.

| Variant | Isometric image 3D | Picture x300 |
|---------|--------------------|--------------|
| T1      | ![Isometric Image](image1) | ![Photographic Image](image2) |
| T2      | ![Isometric Image](image3) | ![Photographic Image](image4) |
| T3      | ![Isometric Image](image5) | ![Photographic Image](image6) |
| T4      | ![Isometric Image](image7) | ![Photographic Image](image8) |
| T5      | ![Isometric Image](image9) | ![Photographic Image](image10) |
The quality of surface treatment in preparation for adhesive bonding was determined by means of contact angle measurement and the surface free energy estimation. Table 7 shows mean values (including the standard deviation) of the SFE of 316L steel substrates following mechanical treatment with P320 abrasive tool. The components of the SFE, the polar and the dispersive components of the SFE, are also shown in the Table.

| 316L steel after machining P320 (T2) | SFE [mJ/m²] | Polar component SFE [mJ/m²] | Dispersive component SFE [mJ/m²] |
|-------------------------------------|-------------|-------------------------------|---------------------------------|
| Average value                       | 62.1        | 15.4                          | 46.7                            |
| Standard deviation                  | 1.6         | 1.3                           | 1.5                             |

The results delivered from the conducted study have shown small scatter, which indicates the uniform energy state of adhered surfaces. This is a factor of beneficial effect on the strength of adhesive joints.

Figure 3 shows the effect of cyclic thermal loading on the strength of adhesive joints. The standard deviation values show the scatter of results. Adhesive joints bonded with Hysol 9466 and Hysol 9489 adhesive compositions were subjected to strength tests. The results from the tests [12, 13] appear to imply that thermal loading does affect adhesive joint strength. Past studies were performed for 200 thermal shock cycles.

Our tests specifically show that the strength of adhesive joints tends to decrease after thermal loading. The drop in strength values of adhesive joints was observed in both adhesive compositions, Hysol 9466 and Hysol 9484. The samples bonded with the former exhibited lower joint strength by 19% after having been subjected to 500 cycles of thermal shock (TS), compared to samples prior to thermal loading. Samples bonded with the latter adhesive, Hysol 9484, subjected to identical thermal loading showed an even higher drop in strength, which was lower by to 28% than in samples not loaded thermally. It was furthermore observed that after 500 loading cycles, the strength characteristics of joints cease to deteriorate. It was found that thermal shocks (TS) increase the scatter of results of Hysol-9466-bonded joints. Fully cured, this adhesive composition is characterised by higher rigidity than Hysol 9484, which may have been the reason for the observed dependency. Hysol 9484 did not show significant increase in scatter of results.
4. Conclusions
Based on the performed tests, it is possible to draw the following major conclusions:

1. In the 316L steel substrate material, sand-blasting generates a more desirable surface texture than the other surface treatments analysed in tests.
2. The strength of adhesive joints bonded with Hysol 9466, subjected to surface treatment variant T2 is 100% higher compared to the untreated samples (T1).
3. Insignificant scatter of results in the values of the surface free energy and its components determined in tests indicates that the energy state of the surface of adherends following treatment variant T2 is uniform.
4. The 316L steel adhesive joint samples bonded with Hysol 9466, after mechanical treatment with P320 abrasive tool, that were subjected to cyclic thermal loading (500 cycles) show a decrease of 19% in mean shear strength compared with the samples prior to thermal loading.
5. The most considerable drop in the mean shear strength of the thermally loaded adhesive joints (500 cycles) was noted in the samples bonded with Hysol 9484, whose strength was 28% lower than the strength of the unloaded samples.

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