TESTING OF ADHESIVE JOINTS DURING CURING AS A FUNCTION OF TEMPERATURE AND TIME

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Abstract

This article focuses on the effect of curing temperature on the strength of adhesive joints used in vehicle prototypes. Testing the influence of curing temperature on the adhesive joint was carried out on samples of aluminum alloy EN AW 5754, with the adhesive surface prepared by abrasive blasting. Samples of aluminum alloy were bonded using three types of two-component adhesives. Two types of adhesive joints are addressed here. Firstly, adhesive joints cured at +20 °C for 168 hours, and secondly adhesive joints cured at +80 °C for 1 hour. The samples are tested using a tensile test, and the resulting data is plotted in graphs in the experimental section and compared. The achieved results of the experimental activity are discussed in the conclusion.

Keywords: Glued joint, adhesives, adhesives, lap joint, aluminum alloy, shear load

1. INTRODUCTION

Material bonding represents a key technological process, the importance of which is growing in a wide range of industrial sectors. The topic of testing bonded joints during curing, depending on temperature and time is becoming increasingly relevant, as the adhesion between bonded surfaces plays a crucial role in achieving the desired functionality and reliability of the final product. As numerous studies have shown, the strength of the bonded joint is influenced by many factors, among which key are surface treatments of materials [1], surface cleanliness, and uniform adhesive application [2,3]. Additionally, the influence of the surrounding environment on the strength of the bonded joint, has also been observed. Temperature therefore also plays another crucial role in the strength of the bonded joint.

This research focuses on analyzing the relationship between temperature, curing time, and the ultimate strength of adhesive bonds. While adhesive bonding technology offers numerous advantages such as minimizing the impact of thermal stress on the bonded material, resistance of the bonded joint to environmental factors, ease of bonding different, commonly incompatible materials, etc., designing and implementing an adhesive bond with guaranteed mechanical properties is not a simple task. It is essential not only to rely on data provided by adhesive manufacturers, but also to conduct systematic testing, which includes testing according to standards.

The aim of the work is to conduct a detailed analysis of the curing dynamics of two-component adhesives under various temperature and time conditions. This issue is of fundamental importance for the optimization of adhesive technologies in manufacturing processes and ensuring higher quality and reliability of products in practice. Experimental results will be presented in this work and factors influencing the properties of bonded joints will be evaluated, with an emphasis on the practical application of findings and their potential use in the production process.

2. EXPERIMENTAL

At the beginning of the experiment, an experimental matrix was established, specifying the types of adhesives and curing temperatures for the bonded joints. In order to ensure informative measurement values and reproducibility, 7 samples were produced for each type of bonded joint. In total, the strengths of 3 types of adhesives were compared at temperatures of +20 °C and +80 °C.

Specification of adhesives:

DP 460 3M Scotch-weld –This is a two-component epoxy adhesive, with a modified epoxy as the adhesive and a modified Amine as the hardener. The workability of the adhesive is 1 hour. Curing at 20 °C takes seven days.

7260 B/A FC, 3M Scotch-weld – This is a fast-curing two-component epoxy adhesive. It contains glass beads with a size of 0.3 mm to achieve precise adhesive thickness during bonding. The workability of the adhesive is 2 hours. Final curing of the adhesive occurs after seven days.

LOCTITE EA 9497, Henkel Adhesives – This is a two-component epoxy adhesive that cures at room temperature. The workability time is 3-4 hours. Final curing of the adhesive occurs after seven days.

Surface preparation of samples and surface roughness measurement.

The surface of the selected materials was prepared using the blasting method in the blasting box SBC 420. Brown fused alumina F090 was used as an abrasive. The average surface roughness value - Ra [μm] was further checked for each sample. For the material EN AW 5754, the measured value of Ra was 3.14 ± 0.15 μm. The measurements were taken using a Mitutoyo SJ 410 device (**Figure 1**). After mechanical cleaning using the blasting method, the bonding surfaces were cleaned at the bonding location using Isopropyl alcohol, which ensured the cleaning and degreasing of the surface before bonding. Subsequently, reference samples were also created without surface treatment, only cleaned using isopropyl alcohol.

Figure 1 Roughness inspection of the bonded surface using Mitutoyo SJ 410 device

The bonded area of the sample had a length of 12.5 ± 0.25 mm, a width of 25 mm ± 0.25 mm, and a material thickness of 5 mm, as shown in **Figure 2**. Therefore, the total length of the bonded test sample was 187.5 mm. These dimensions were based on the standard ČSN EN 1465.

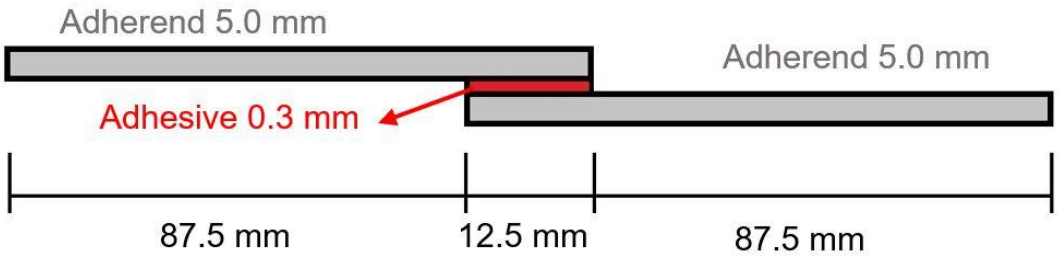

Figure 2 Detail of the selected overpayment of the bonded joint

Device for precise gluing

For repeatability of the gluing process, a fixture (**Figure 3**) was used, ensuring precise bonding of samples and the correct adhesive thickness as specified in the technical data sheet at 0.3 ± 0.05 mm for all samples. This fixture provided fixation during the heat curing process, as it was preheated to the desired temperature before the samples were placed.

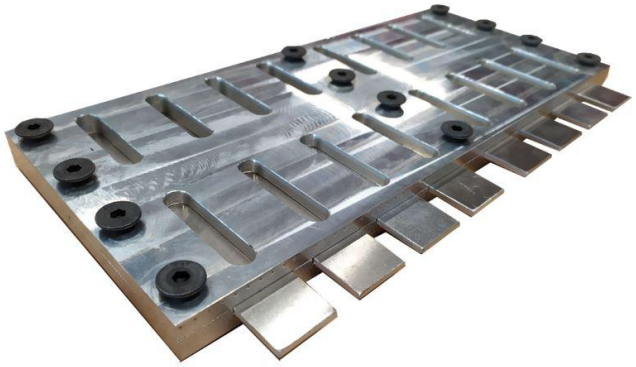

Figure 3 Device for precise gluing

Tensile test

Tensile test according to the standard ČSN EN 1465 (668510) - Determination of shear strength under tensile loading of overlapped bonded assemblies was performed on a tensile testing machine SHIMADZU AG-X plus 50 kN. Samples were tested using tensile strength test, where the sample was clamped between two vertical grips.

3. RESULTS

Within a large number of samples and tensile tests conducted, column graphs were created for clarity, containing the measurement deviation derived from individual tensile test measurements on 7 samples for a given type of adhesive.

Adhesive joint DP 460

During the tensile test, the adhesive joint at +20 °C exhibited the highest tensile strength value of 26.0 MPa, an average tensile strength value of 24.9 MPa, and a standard deviation of measurements of 0.95 MPa. The average elongation value was around 1.34 mm. The manufacturer-specified average value is 32.0 MPa.

Figure 4 shows the results of the tensile test of the bonded joint with the adhesive cured at +80 °C for 60 minutes. The maximum tensile stress value measured was 25.3 MPa, the average tensile stress value was 22.9 MPa, and the standard deviation of the measurements was 1.9 MPa. The average elongation value was around 0.80 mm. The manufacturer's reported average value is 18.0 MPa.

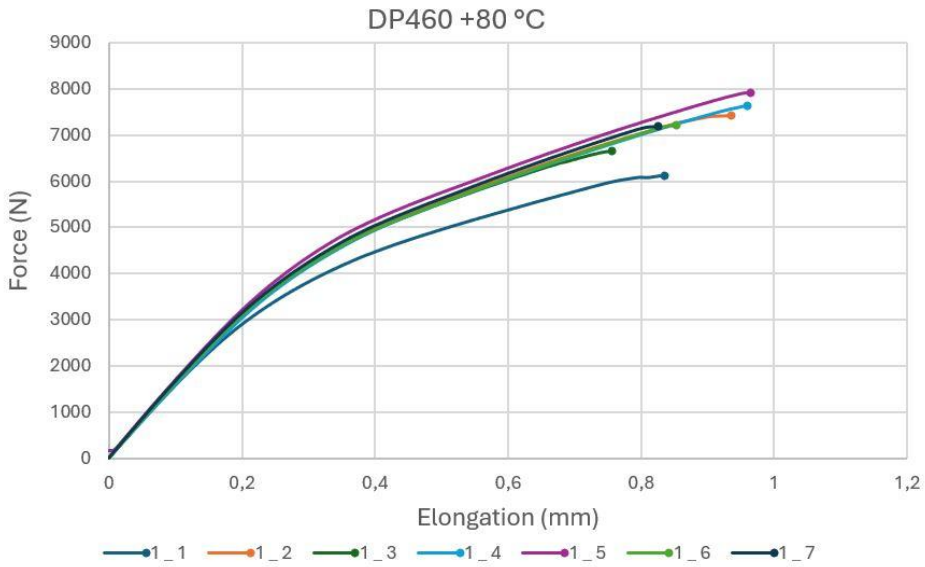

Figure 4 Measurement profiles of individual samples of adhesive joint DP 460 at +80 °C

Comparison graph of stated and measured values for a bonded joint using the 3M DP 460 two-part adhesive. From the graph, as shown in (**Figure 5**), a decrease in the strength values of the bonded joint is noticeable when cured rapidly at +80°C for 1 hour. Interestingly, lower strength values of the bonded joint are achieved than those stated by the manufacturer at +20°C, compared to achieving higher strengths when cured at +80°C. This phenomenon will be discussed in conclusion.

Adhesive joint S-W 7260

At +20°C, the bonded joint exhibited the highest tensile strength value of 28.0 MPa, an average tensile strength value of 23.5 MPa, and a standard deviation of measurements of 1.9 MPa. The average value of the elongation of the bonded joint was around 0.91 mm. The manufacturer's stated average strength value of the bonded joint of the given material is 33.0 MPa.

Interesting values were achieved by the bonded joint at +80°C, where the samples showed the highest tensile strength value of 22.6 MPa, an average tensile strength value of 21.1 MPa, and a standard deviation of measurements of 1.7 MPa. The average value of elongation was around 0.86 mm. The manufacturer's stated average value is 20.0 MPa. Here, we were already able to reach the manufacturer's stated values within the range of measurement errors.

Adhesive joint EA 9497

Adhesive joint using adhesive EA 9497 exhibited the highest tensile strength value of 17.4 MPa at +20 °C, with an average tensile strength value of 15.7 MPa and a standard deviation of measurements of 1.0 MPa. The average elongation value was around 0.36 mm. The manufacturer's stated average value is 15.0 MPa.

The adhesive joint at +80 °C showed a maximum tensile strength value of 14.5 MPa, an average tensile strength value of 13.0 MPa, and a standard deviation of measurements of 0.7 MPa. The average elongation value was around 0.33 mm. The manufacturer's stated average value is 14.0 MPa.

4. CONCLUSION

The measured data from pull tests for individual adhesives and materials are clearly displayed in (**Figure 5**). Results of strength measurements of different types of bonded joints are compared with the values provided

by the manufacturer. The graph shows the average strength value, and the measured standard deviation determined from 7 measurements of the same type of sample.

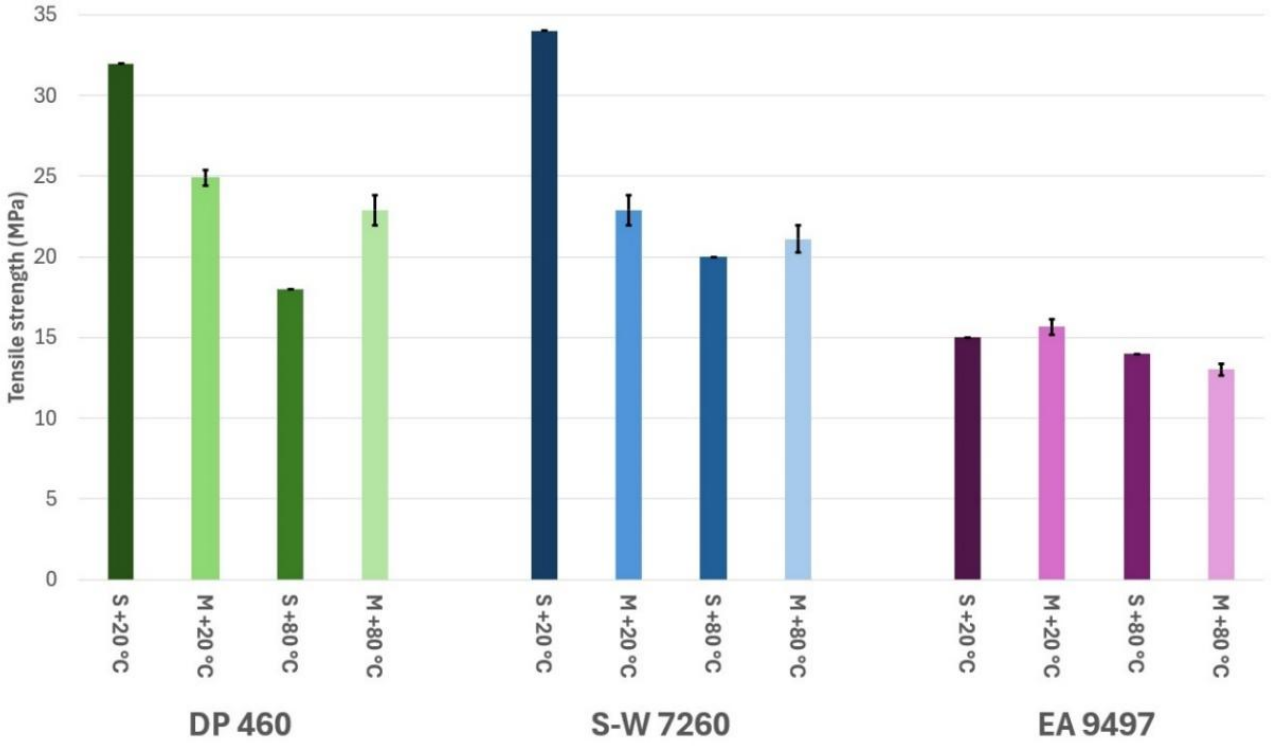

Figure 5 Summary of tensile test results

Designation „S“ - Specified by the manufacturer, „M“ – Measured

In the case of bonded joints using adhesives DP 460 and S-W 7260, considering the strength values of the bonded joints and the standard deviations of two different adhesive materials, it is possible to conclude a very similar almost identical strength of these adhesives under the given conditions. For adhesives DP 460 and S-W 7260, the strength of the bonded joint at a temperature of +20 °C and a curing time of 168 hours is lower than what the manufacturer states. At a temperature of +80 °C and a curing time of 1 hour, the strength of the bonded joint is higher than what the manufacturer states. With adhesive EA 9497, the opposite effect occurs, where at a temperature of +20 °C and a curing time of 168 hours, the strength is higher than what the manufacturer states. At a temperature of +80 °C and a curing time of 1 hour, the strength of the bonded joint is lower than what the manufacturer states. **Table 1** shows a comparison of measured strength values of the adhesive joints in tensile test with the strength values provided by adhesive manufacturers.

Table 1 Comparison of the achieved strength values of adhesive joints with values from technical data sheets of adhesive manufacturers

Glue	Aluminium alloy (MPa)			
	Specified +20 °C (168 hours)	Measured +20 °C (168 hours)	Specified +80 °C (1 hour)	Measured +80 ° (1 hour)
DP 460	32.0	24.9	18.0	22.9
S-W 7260	33.0	23.5	20.0	21.1
EA 9497	15.0	15.7	14.0	13.0

The decrease in strength in the measured values between +20 °C and +80 °C is on average 2.4 MPa, compared to the time savings of the curing time of the adhesive joint, which is recommended at +20 °C approximately 168 hours, it is necessary to take into account the decrease in strength of the adhesive joint versus the rapid time savings, which is 167 hours. This can significantly affect both the product price and the speed of production of the part. If the technology allows for the adhesive joint construction to cure at +80 °C, this represents a considerable acceleration of the production process.

Since the manufacturer does not specify the surface treatment used for individual materials, we consider that the differences in measured strengths and stated adhesive joints are due to different surface preparation methodologies before bonding. Therefore, it is advisable to directly verify the surface preparation methodology of the material through tensile testing, which plays a crucial role.

As a further development direction in adhesive joints for composite materials, it would be appropriate to study the influence of the ambient temperature on the strength of the adhesive joint, when the suitability for use in bonding automotive components has been confirmed, which can reach temperatures higher than +20°C, where the different compositions of these types of structural adhesives may affect the mechanical properties.

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