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Research Article

Experimental Investigation of the Effect of Curing Pressure on Strength of Single Lap Adhesive Joints

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ABSTRACT

It is a common method to apply a certain pressure to the bonded surfaces while bonding. In this way, it is ensured that the adhesive covers all surfaces and the air in the adhesive is evacuated. However, the effects of pressure changes on the curing process and mechanical properties, especially in adhesives that harden by chemical reaction, are an issue that should be handled with numerical data at a scientific level. When the literature is examined, there are limited studies on the effects of curing pressure on mechanical properties of adhesive joints.

In this study, the effect of curing pressure on bond strength in adhesive joints was investigated experimentally. Single lap adhesive joints were manufactured using steel plate and DP460 epoxy adhesive. At this stage, bonding joints with different curing pressure conditions were obtained by placing unit weights on the adhesive area. The samples fabricated five different curing pressure values were subjected to tensile tests. In consequence of the experiments, a reducing in the joint endurance was observed after a certain pressure value. When the test results are reviewed, by increasing the curing pressure from 0.22 N/cm^2 to 0.44 and 0.66 N/cm^2, the decrease in the failure loads was calculated as 0.8% and 6.8% respectively, while the decrease in the values of 0.88 and 1.1 N/cm^2 was computed as 20.9% and 49% respectively. Thus, it has been understood that the pressure applied to the adhesive area affects the curing process significantly, and must be taken within certain limits.

1. INTRODUCTION

Adhesive joints are widely used for joining due to their superior properties such as distributing the load over a wider area than the other connection types, not requiring holes, obtaining lightweight structures and having superior fatigue resistance [1]. Some of the factors affecting the performance of the bonded joints are; surface treatments applied to the bonded materials, the ambient temperature in which the adhesive joint is employed, the residual thermal stresses owing to the difference in the thermal expansion coefficient of the adhesive and the bonded materials, curing conditions, particles reinforcement to adhesive and joint geometry [2-3].

In bonding joints, the curing of the adhesive is a very significant issue that directly influences the strength of the joints. A poorly cured adhesive is a critical issue for the aerospace and automotive industries as it can jeopardize the integrity of adhesive joints [4]. Adhesion and cohesion forces are effective in bonding joints. Adhesion forces occur between the adhesive and adherent, the two surfaces are held together by interface forces. Cohesion occurs within the adhesive itself. In cohesion, the mass components that make up the adhesive are held together by chemical or physical forces. High-strength adhesives used in engineering applications were harden by chemical reaction. This chemical process in which adhesives obtain their strength is called curing.

At the end of the curing process, chemical bonds are formed between the adhesive molecules and the necessary strength is gained. Since the adhesion properties of adhesives are very delicate to environmental circumstances, the load bearing capabilities of the adhesive joints are influenced by the pressure and temperature applied during curing operation. One of the adhesive types that cures by chemical reaction is epoxies. Epoxy adhesives can be in three different forms as one-component, two-component liquid and film. The two-component ones consist of an epoxy adhesive and a hardener, when both are mixed, covalent bonds are formed to become a thermoset polymer [6]. Covalent bonds are the primary bonds that hold atoms together. It is very strong in covalent bonds such as ionic and metallic bonds. Therefore, the strength values of adhesives cured by chemical reaction are considerably higher than adhesives cured by physical change [7].

Pure epoxy molecules at room temperature do not normally react with each other. Types of chemicals added to epoxy to provide network formation; it is divided into two parts as curing agents and catalysts. Curing agents, sometimes called hardeners, are added to epoxies in considerable quantities and react with the epoxy to build a cross-linked network. Cured resins become insoluble, used to join almost all types of materials (metals, glass, ceramics, wood, composites). They are chemically resistant and have striking dielectric properties. Regarding the curing method, adhesives can be divided into three parts. These; curing at room temperature, curing at average temperature (up to 120 °C), curing at elevated temperatures (up to 250 °C). The mechanical properties of structural epoxy adhesives are strongly dependent on the curing temperature [8]. Curing times are directly related to the heat implemented to the adhesive. Curing time can be reduced at higher temperatures. For epoxy adhesives, curing times at room temperature are up to 24 hours, while curing times decrease with increasing temperature values. The curing times of DP460 Epoxy adhesive, which was also used in this experimental study, are recommended as 24 hours at 23 °C, while it can be reduced to 1.5 hours at 49 °C and 1 hour at 60 °C [9].

There are studies on the effect of curing temperature and curing times on the adhesion performance of structural and non-structural adhesives [10-17].

It is a common method to apply a certain pressure to the bonded surfaces while bonding. In this way, it is ensured that the adhesive covers all surfaces and the air in the adhesive is evacuated. However, the effects of pressure changes on the curing process and mechanical properties, especially in adhesives that harden by chemical reaction, are an issue that should be handled with numerical data at a scientific level. When the literature is examined, there are limited studies on the effects of curing pressure on mechanical properties of adhesive joints.

It has been stated that increasing the applied pressure during curing of cylindrical bonding joints produced using inflexible epoxy adhesive causes the residual stresses which reduces the joint performance. The curing process was done in an autoclave at different pressures [4]. The hydrostatic pressure applied to the adhesive injected between the shaft and the hub increased the performance of the joint in the shaft-hub connections [18]. In single-lap adhesive joints produced using flexible adhesive, the increase in curing pressure improved the joint performance [19].

In this experimental study, the effect of the curing pressure applied to the adhesion area on the tensile strength of the joints was examined in single-lap joints produced using inflexible epoxy adhesive. The purpose of the work is to show the effect of mechanically applied curing pressure on the on the strength of single-lap joints. The motivation of the study is that the data sheets prepared by the manufacturers of epoxy-type adhesives do not include detailed information on the effects of mechanically applied pressure on the adhesion performance.

2. MATERIAL METHOD

2.1. Adhesive and adherend materials

In the experimental study, AISI 304 steel sheet with a thickness of 2 mm was employed as adherend parts. DP460 epoxy manufactured by 3 M Company (St. Paul, MN, USA), was used as adhesive. DP 460 is a two-component and widely used structural adhesive. Mechanical properties of bonded and adhesive materials are presented in Table 1 [20-21].

TABLE I
MATERIAL PROPERTIES OF THE ADHERED AND ADHESIVE

Properties	AISI 304	DP460
Ultimate tensile strength, σ_t (MPa)	515-720	44.6 ± 1.2
Yield strength, σ (MPa)	210	38.4 ± 1.1
Modulus of elasticity, E (MPa)	190.000	1984 ± 43
Poisson's ratio, υ	0.29	0.37
Ultimate tensile strain, ε_t (%)	≥ 50	4.7

2.2. Fabrication of the tensile test samples

In order to produce single-lap adhesive joint samples, pieces of 130x25x2 mm were cut from AISI 304 plates by laser cutting method. The joint geometry and dimensions used in the study are shown in Figure 1.. Surface preparation process is necessary for adhesive joints to exhibit superior performance. Surfaces to be adhesive applied were eroded with P180 grade emery paper to take away pollutants and to roughen. Subsequent to this, abraded surfaces were washed with liquid detergent under tap water. Following this, the washed parts were dried with a cloth towel. The bonded surfaces were purified via acetone and washed by tap water again. Bonding surface conditioning procedures were completed by drying the adherends with paper towel and finally heated air.

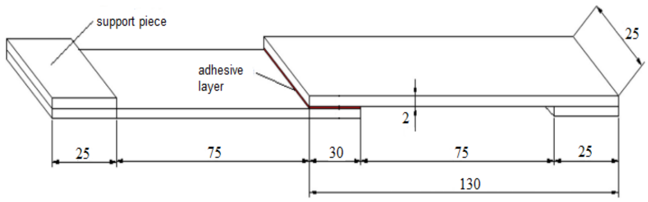

Figure 1. Test specimen geometry

Two-component epoxy adhesives consisting of hardener and epoxy resin were supplied in 50 ml tubes. The applicator was used to discharge equal amounts of hardener and epoxy resin. After the adhesive is poured into a clean container, the hardener and epoxy resin were blended using a wooden pin, in this way, the adhesive material was prepared (Figure 2).

Figure 2. Adhesive pouring applicator and adhesive

To fabricate test samples of single-lap joints in reference to joint geometry, adhesive was implemented on the bonding area and the specimens were placed into the pattern (Figure 3, Figure 4). Three samples were produced at once in the mold.

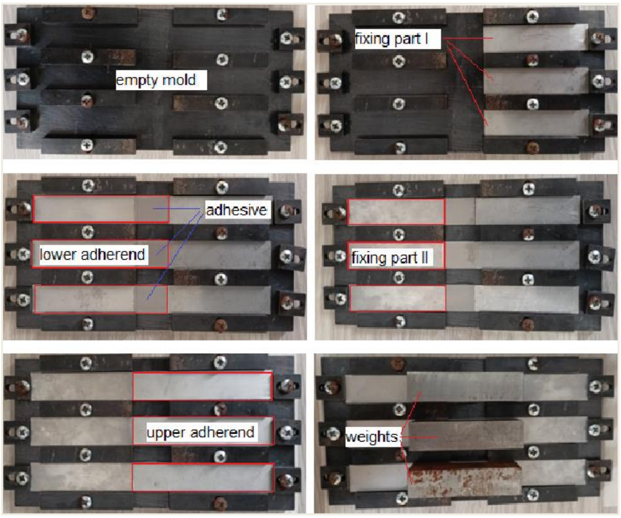

Figure 3. Manufacturing steps of specimens

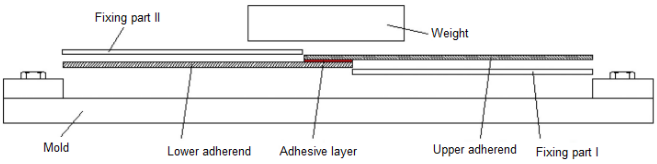

Figure 4. The schematic diagram of the mold

In this study, the effect of the pressure applied to the adhesive layer during the curing process on the tensile strength of the joints were examined. Pressure was applied to the adhesive layer using steel rectangular prism-shaped unit weights. Accordingly, in the bonding region; different pressure values were obtained by using one, two, three, four and five weights, respectively. The pressure values used in the study were calculated as 0.22 N/cm^2, 0.44 N/cm^2, 0.66 N/cm^2, 0.88 N/cm^2, 1.1 N/cm^2 according to the weights used. As an example, the molding step of samples produced at 0.88 N/cm^2 and 0.44 N/cm^2 curing pressure using four and two weights is shown in Figure 5. Other curing pressure values were obtained with the same method using different numbers of weights. The samples were kept at the desired curing pressure in the mold for 24 hours. Then, after the samples were carried away from the mold, they were coded and stored in a protective container until the experimental stage.

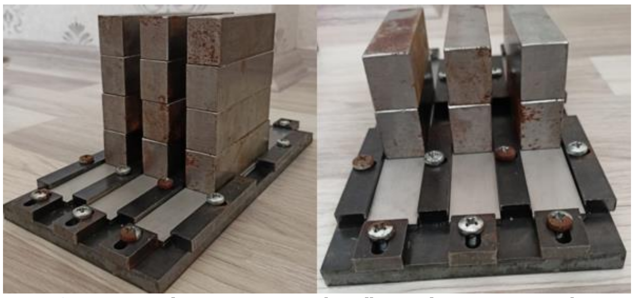

Figure 5. Applying pressure to the adhesive layer using weight

In the study, samples were produced without applying pressure to the adhesive coating. However, as a result of the experiments, it was understood that the adhesive did not spread well on the surface and the adhesive thickness was not stable in the samples, so the samples prepared without applying pressure were excluded from the test program. Therefore, it has been understood that a certain amount of pressure must be applied to the adhesive area for fully spread the adhesive on the surface and to obtain an even adhesive thickness along the adhesive line.

2.3. Tensile testing of the joints

A series of tensile test were implemented to decide the effects of pressure variation in the adhesive layer during the curing phase.

Tensile tests were carried out under laboratory conditions at a tensile speed of 1 mm/min (Figure 6). Sample types were determined according to the curing pressure (Table 2). Experiments were made using five joint types. To raise the reliability of tests, four specimens were prepared for each specimen type, in all, 20 specimens (5 joint type x 4: 20).

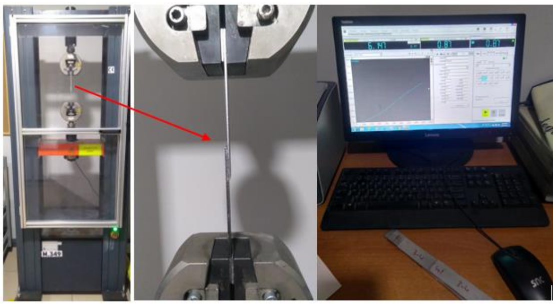

Figure 6. Tensile test application

Figure 7, shows 5 test samples produced for the P-1 sample type.

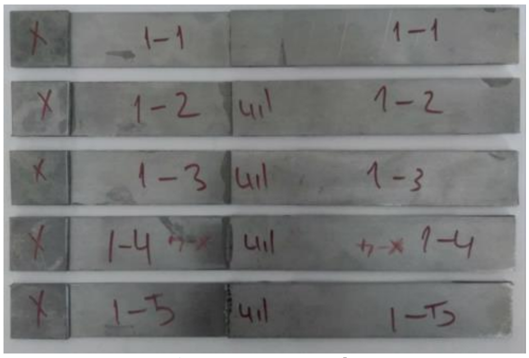

Figure 7. SLJs samples

The samples were carefully monitored while testing and the maximum damage loads and maximum tensile strains of single-lap joints were reported. The adhesive thickness of all samples removed from the mold was measured in the range of 0.05 mm-0.1 mm. The curing process of the pressure applied samples was completed in 24 hours under room temperature conditions (22 °C). Boundary conditions for single-lap adhesive joints were determined in accordance with the tensile test conditions (Figure 8).

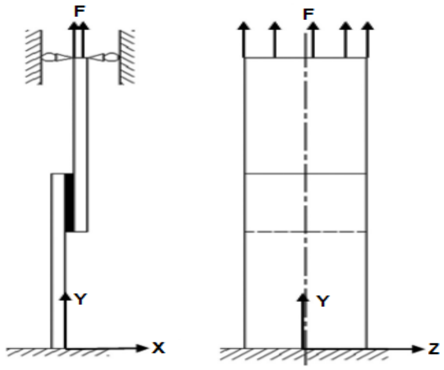

Figure 8. The boundary setup of the joints

TABLE 2
JOINT TYPES

Joint type	Curing pressure(N/cm^2)
P-1	0.22
P-2	0.44
P-3	0.66
P-4	0.88
P-5	1.1

3. RESULTS and DISCUSSION(Helvetica 10p Bold)

The experimental tensile failure loads of single-lap joints are obtained according to the curing pressure (Figure 9).

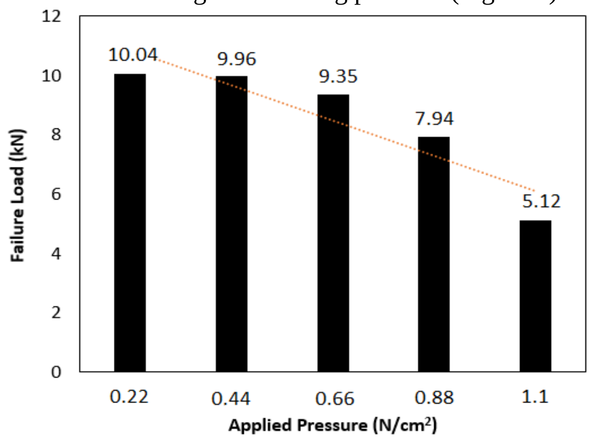

Figure 9. Failure load variation according to curing pressure

When the results are examined, the failure loads of the Single-lap joints, at the pressure values of 0.22 N/cm^2, 0.44 N/cm^2 and 0.66 N/cm^2 are close to each other as 10.04 kN , 9.96 kN and 9.35 kN, respectively. However, when the pressure values were increased to 0.88 N/cm^2 and 1.1 N/cm^2, a significant decrease was observed in the failure loads (At pressure values of 0.88 N/cm^2 and 1.1 N/cm^2, the failure loads were measured as 7.94 kN and 5.12 kN, respectively). When the adhesion theories in the literature are examined; the mechanical adhesion theory is based on the principle that the adhesive penetrates the pores on the surface of the material and is physically locked there [19]. Adsorption theory, developed by Sharpe and Schonhorn, suggests that adhesion occurs as a result of the formation of interatomic and intermolecular forces at the interface, provided that good contact between the base material and the adhesive is maintained [20]. Adsorption theory is still the most widely accepted and applicable theory in adhesive science. Therefore, it can be said that applying pressure to the adhesive layer increases the bond performance, as it allows the adhesive to spread better on the surface and penetrate into the pores, according to these theories. However, it is seen in this study that this approach can be correct up to a certain pressure value and that increasing the pressure further reduces the joint strength. Accordingly, with an rise of 5 times the curing pressure, the average tensile failure load decreased by 50 %.

During the curing of the adhesive joints, residual thermal stresses occur because the adhesive and the bonded materials have different thermal properties. Especially in bonding processes at high temperatures, these stresses increase even more. At this stage, it can be thought that the pressure applied to the adhesive layer will affect the residual stresses that occur during curing. It is a research question, whether the pressure applied during curing will affect the joint strength positively or negatively. Another factor is the curing temperature. When the literature is examined, in a study using a flexible adhesive (SBT 9244), the curing was made using a hot press at 145 °C at two different pressures as 0.1 MPa and 0.5 MPa, and the effect of the curing pressure was experimentally measured [16]. Accordingly, it has been determined that the increase in pressure increases the joint strength. In another study, cylindrical bonding joints were formed by using steel as the bonded material and DP 460 (3M, USA) as the adhesive [1]. In the study, the effect of curing pressure on the joint strength was investigated experimentally by performing tensile tests. As a result of the experiments, it was determined that increasing the curing pressure decreases the joint strength. During the production of the test samples, the curing temperature was 80 °C. In a experimental study, while explaining the effect of curing pressure on the joint strength, they stated that the pressure increased the heat transfer coefficient of the air in the autoclave where the curing was carried out, and as a result, the curing operation accelerated and the adhesive temperature increased [1]. It was stated that as a result of the increase in the adhesive temperature, the thermal residual stresses increased and the joint was damaged earlier.

As stated in the literature, residual stresses negatively affect the joint performance. It is understood that the pressure applied to the adhesive layer during curing causes residual stresses and reduces the bond strength. It is known that the pressure applied to the adhesive causes the adhesive to completely wet the surface and establish a very good contact with the adhered material. However, after providing the required pressure value as seen in this study, the effect of further increasing the pressure on the strength was reduced.

Two-component epoxy adhesives start to react at ambient temperature after the two components are mixed. The reaction rate is greatly affected by the reaction temperature. For every 10 °C increase in temperature, the reaction time is doubled. Full cure times at ambient temperatures for two-component systems range from a few minutes to several days. The decrease in crosslinking levels results in low cohesion strength. Similarly, in this study, it is thought that the curing pressure change affects the bond strengths by changing the reaction rate.

At the end of the tensile tests, load displacement graphs were obtained (Figure 10). The load displacement graphs have been prepared by taking into account the data of the test sample with the largest failure load in the same test group. Accordingly, the displacement amount decreased with the increase of the curing pressure value.

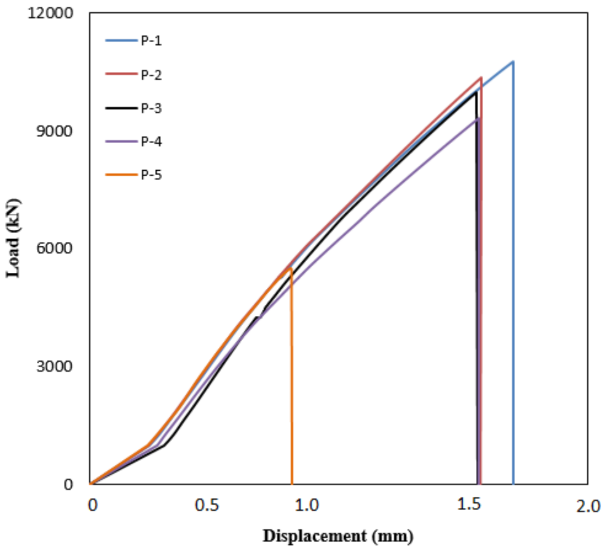

Figure 10. Load displacement graphs according to curing pressure

4. CONCLUSIONS and SUGGESTIONS

In this study, the effect of the curing pressure applied to the adhesive area on the strength was investigated by performing tensile tests. The general results obtained experimental study are given below:

In cases where two-component epoxy adhesives that can be cured at room temperature are used, pressure must be applied to the bonding area during curing in order to spread the adhesive over the entire surface and to obtain a homogeneous adhesive thickness. However, it should be noted that after a critical pressure value, there is a decrease in joint strength due to residual stresses.

In the experimental study, the highest average damage load was obtained with a value of 10.04 kN at 0.22 N/cm^2 curing pressure. Failure loads were close to each other at 0.22 N/cm^2, 0.44 N/cm^2 and 0.66 N/cm^2 curing pressure values. An important decrease in bond strength was seen at the curing pressure values of 0.88 N/cm^2 and 1.1 N/cm^2.

By increasing the curing pressure from 0.22 N/cm^2 to 0.44 and 0.66 N/cm^2, the decrease in the damage load value was calculated as 0.8% and 6.8%, respectively, while the decrease in the values of 0.88 and 1.1 N/cm^2 was computed as 20.9% and 49%, respectively. Therefore, the curing pressure affects the joint strength negatively after a certain pressure range. According to this study, the recommended curing pressure range is 0.11 N/cm^2 - 0.66 N/cm^2.

This study does not show how curing pressure affects the chemical mechanics/interaction in the adhesive. It would be beneficial to conduct studies on this subject in future.

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