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Mechanical Properties of the Combined Single Lap-Shear Connections with Graphene Doped Epoxy Adhesives

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Abstract – In this study, it is aimed to strengthen epoxy with graphene as an adhesive, because of recently the outstanding properties of graphene. For this purpose, the effects of graphene usage on shear strength of modified epoxy-based adhesives were examined. The shear strengths of the created bondings are investigated. For this goal, Al 2024-T3 sheet was used as a bonding material. While determining the mechanical properties of graphene-doped epoxy based adhesives, the experiments were carried out in accordance with ASTM D1002-10, single lap sheared connection. In order to carry out these processes, surface treatment of aluminum plates, formation of adhesive materials, bonding process and drawing casting of lap-shear samples to measure shear strength were applied respectively. Graphene were used in 0.25, 0.50, 0.75, 1% by weight. Five (5) samples were produced from each sample for the reliability of the experiments. Morphological structure and diameters of the formed samples were analyzed by scanning electron microscopy (SEM) images. The shear strength of the epoxy-based adhesive by adding Graphene resulted in an increase when compared to neat epoxy. On the other hand, the ultimate shear strain of the modified adhesives decreased by adding the nano particles into the epoxy resin.

Keywords – Graphene, Epoxy, Shear Strenght, SEM, Lap-Shear

I. INTRODUCTION

Epoxy resins have a wide range of excellent physical and chemical properties which makes them essential among the current technologies and for the development of new technologies. Owing to their strong adhesion to a variety of treated or untreated metal surfaces[1], epoxy-based adhesives are extensively employed in many industries especially in aircraft industry, in order to bond to different materials' substrates [2]. Bonding of the parts made by similar and/or dissimilar materials is widely used industrial process [3]. Adhesive bonding is widely used in many areas of industry from the aerospace to medical applications [4]. The mechanical or physical property of the epoxy resin can be improved by adding the several metallic nano particles. However the mechanical properties of the epoxy matrix can be negatively affected for higher weight ratio of additive-epoxy. It is expected from the additive materials to cause reinforcement or enhance any superior properties of composite materials. But the higher weight ratio of the additives can be negatively effect to composites because of inhomogeneity. In addition, higher additive ratios can effect the lower adhesion between the matrix and additive negatively [5]. The mixture of the epoxy resin and its curing agent exhibits as a brittle behavior after the mixture cured. But some applications of these types of adhesives are expected to have ductile behavior especially the metal-metal joints [6]. Adhesive joints can be used at various temperatures. Durability and strength at extreme temperatures have always been a major limitation of adhesives. When repairing some electronic parts by using epoxy based adhesives they can be conserved from the negative effects of higher temperatures of soldering or welding [7].

II. MATERIALS AND METHOD

In this study, 1.62 mm thick Aluminum 2024-T3 sheet was supplied by Turkish Aircraft Industry as the adherend, MGS-L285 lamination resin and MGS-H285 curing agent as adhesive, graphene as additives. Selected lamination resin and curing agent were used for bonding Al-2024-T3 layers. The epoxy resin used for production was diglycidyl ether bisphenol-A epoxy (Momentive-Hexion L285TM) and curing agent (Momentive-Hexion H285TM) was the aliphatic amine. Graphene were purchased from Nanografi Company. Properties of the graphene were smaller than18 nm thickness and 300 m^2/g specific surface area.

A. Preparation of the Adhesives and Samples

The Al 2024-T3 sheet adherends were cut according to ASTM D 1002-10 standards (101.6 × 25.4 × 2 mm) (Figure 1) and were pre-cleaned by using deionized water.

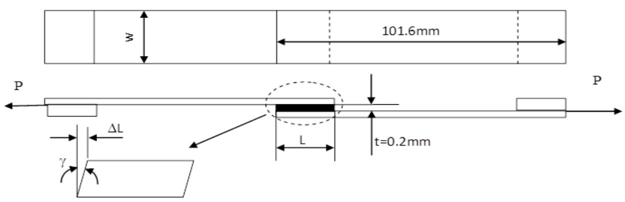

Figure 1. Schematic view of the single lap shear joint sample

In order to achieve a good joint between the adhesive and adherend, Al sheets' surfaces were prepared by chemical etching process and phosphoric acid anodization (PAA), in accordance with ASTM D2651 and ASTM D3933, respectively. As a result PAA an Al2O3 layer came about Al sheets' surfaces. Mechanical locking between the surfaces has been enhanced because of this layer. The surface roughness of the adherends was measured by using Mitutoyo SJ-301 surface tester. Average surface roughness (R_a) of adherends was measured as 1.2 ± 0.12 µm (4 mm cut-off distance) after the surface preparation along and normal to the loading direction [8].

B. Preparation of hybrid nanoadhesives

Flow chart of the preparation of modified nanohybrid adhesives is presented schematically in Figure 2. The weight ratio of epoxy/hardener was 100/40 as manufacturer's advice. The details of the preparation procedure are as follows;

(1) Graphene of given amounts were added to epoxy resin in a beaker.

(2) The epoxy resin and Graphene were stirred using ultrasonic probe homogenizer (Bandelin HD 2200, 20 kHz, 70 W) in ice bath for 30 min. Stirring was discontinued after each 10 minutes in order to avoid the overheating and let mixture to cool down to minimize the damage on nanoparticles due to ultrasonic vibrations.

(3) In order to remove the bubbles and air in to the mixture, it was placed in a vacuum environment at room temperature for 60 min.at 0.6 bar vacuum level.

(4) Curing agent at 40% wt. of epoxy resin was added in to the epoxy.

(5) Modified epoxy adhesive and hardener were mechanically stirred for 10 min at room temperature.

(6) Again the mixture was degassed at room temperature in the vacuum environment for 10 min. to remove air bubbles at 0.75bar.

(7) Modified epoxy adhesives were applied adherend's surfaces.

(8) Adhesion thickness was adjusted by the help of 0.2 shims.

(9) Samples were compressed on to shims at 0.1 MPa and cured in an oven at room temperature for 24 h and then samples were kept for 15 hours at 80 °C for post-curing.

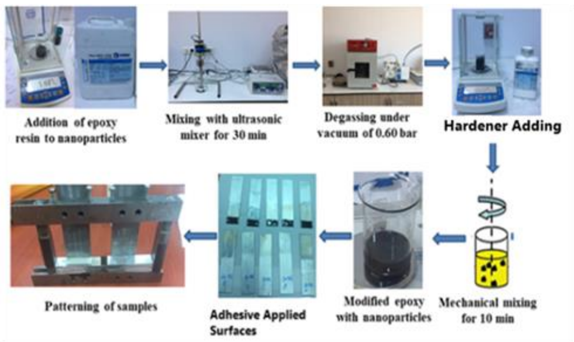

Fig. 2 Schematic flow chart of nanoadhesive preparation.

Weight ratios of graphene into epoxy were selected as 0.15, 0.25, 0.50 and 1 percent. Higher ratios instead of these values have been caused the agglomeration. Table 1 shows the graphene and epoxy resin contents of samples.

Table 1. Contents of the samples in wt %

Sample	Graphene	Epoxy + Hardener
Neat	0	100
025Gr	0.25	99.75
050Gr	0.50	99.5
075Gr	0.75	99.25
1Gr	1	99

C. Tests

Single lap shear tensile tests were carried out according to ASTM D 1002-10 standard. The schematic view of the jointed sample was given in Figure 1.

In order to avoid the bending effect, two supporting parts with identical the sample thickness are used in the grips region. Lap shear tensile tests were performed at crosshead speed of 1 mm/min and at room temperature. For reliability of the results, five samples have the same contents were tested. The mechanical tests were performed by using the Shimadzu AGS-X tensile test machine. The displacements at the jointed region were measured by using Epsilon 3560 model biaxial extensometer.(see Figure 3).

Figure 3.Lap-shear tensile test at Shimadzu AGS-X test machine and using Epsilon 3560 extensometer

The shear stress (τ) at the jointed area of the samples were calculated by the Equation 1.

$$\tau = \frac{P}{b.l}$$

Where P is the applied force, b is the width of the joint, and l the overlapping length. The shear strain (γ) could be calculated as,

$$\gamma = \frac{\delta}{t}$$

Where δ is the displacement at the loading direction and jointed region and t is the thickness of the adhesive. By using the measured shear stress and shear strain, the Shear Modulus of the adhesives were calculated from the Hooke's law in Equation 3.

$$G = \frac{\tau}{\gamma}$$

D. Characterization

SEM images of the synthesized products and fractured surfaces of adhesive joints after single lap shear tests were visualized using a LEO 1430 VP model SEM.

III. Results

A. 3.1. Single Lap Shear Tests

In this study the 5(five) sets of the specimens were tested as seen Table 2. Lap shear tensile test results are presented in Figure 4 for various contents of the specimens. The shear stress-shear strain results are presented in Figure 4 for the specimens which the fixed graphene content was at 0.25 %, 0.50, 0.75 and 1 % wt. ratios.

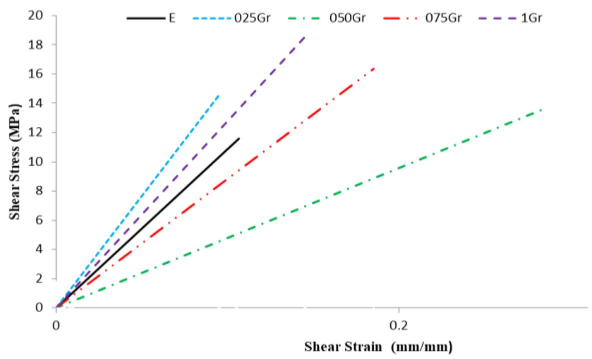

Figure 4. Lap-shear tensile test results for 0.25, 0.50, 0.75 and 1 wt% Graphene and Neat Epoxy.

Table 2. Mechanical Properties of lap-shear adhesive joints

Sample	Shear Modulus (GPa)	Shear Strength (MPa)	Shear Strain (mm/mm)
Neat	101.6	11.75 ±2.92	0.2 ±0.18
025Gr	118.6	13.48 ±1.59	0.081 ±0.020
050Gr	51.12	12.32 ±1.97	0.15 ±0.11
075Gr	87.18	12.38 ±4.61	0.137 ±0.063
1Gr	120.75	17.7 ±2.6	0.148 ±0.048

B. SEM Images of the Fractured Surfaces

SEM images of the fractured surfaces of Neat, 025Gr, 050Gr, 075Gr and 1Gr samples after the shear strenght tests of lap-shear specimens were presented in Figure 5a, b, c, d and e respectively at 10kX magnification. As seen in Figure 5a, the fractured surface of neat epoxy sample is flat and smooth. But with addition to Graphene, the fractured surfaces were roughened and crack progression was observed to be inhibited. It can be seen in Figure 7b that Graphene is obstructed to crack progression by bridging and pull out systems. This effect can be seen for lower ratios of graphene as given in Table 2. Shear strengths of lower additive contents are greater or equal to neat epoxy specimen. But the shear strength of the joints was increased for higher nano particle ratios. The mixtures were tried to prepare homogeneous. However there are few agglomerated regions in Figure 5e by increasing the amount of graphene. The decrease of the strength can be mentioned to because of the inhomogeneity of the mixture.

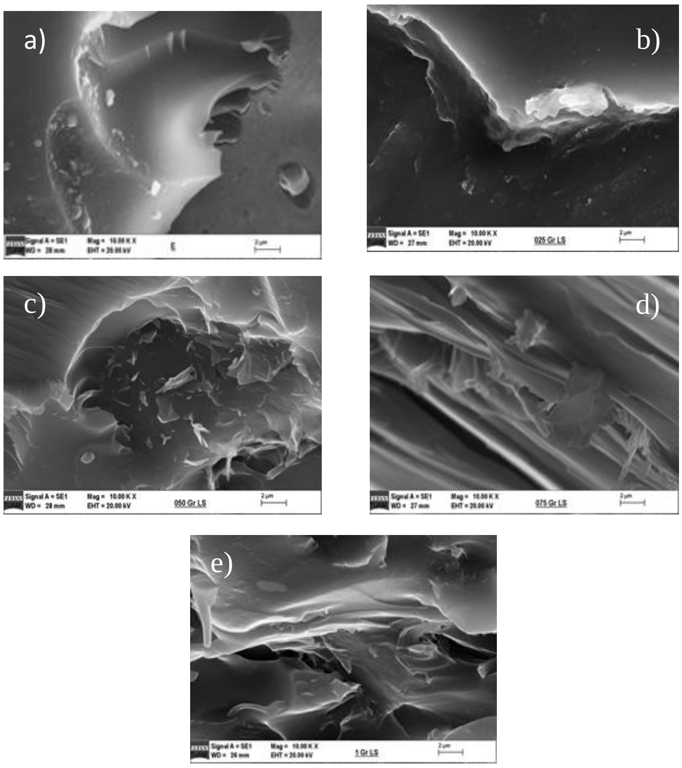

Figure 5. SEM Images of fractured surfaces of a) Neat b) 025Gr c) 050Gr d) 075Gr e) 1Gr samples after Single lap-shear tensile tests

IV. Discussion

In this study epoxy based nano adhesives were produced and obtained their mechanical properties. For obtaining the mechanical properties of the adhesive SLJ specimens were produced and performed to single lap-shear tensile tests. The maximum shear strength were obtained in 1Gr specimen when the minimum shear strength were found in neat epoxy. As a result of investigations, it can be said that adding Graphene to epoxy increases the shear strength of epoxy.

V. Conclusion

In this study, shear strength of the epoxy-based hybrid adhesives were investigated. Graphene was added in to epoxy resin for improving the mechanical properties. Nano particle ratios (in % weight) were selected as 0.25, 0.50, 0.75 and 1 the Graphene. Results were compared with the neat epoxy adhesive. First, effects of Graphene on to shear strength of the epoxy adhesive were obtained. Later SEM images were evaluated.

In order to obtain the shear strength of epoxy adhesive, Single Lap Joint (SLJ) Tensile Tests were performed. Shear strength of Neat Epoxy SLJ was obtained as 11.75 ±2.92MPa while the maximum strength for 17.7 ±2.6 MPa for 1% Graphene added specimen. The shear strength of the produced epoxy-based adhesive by using Graphene resulted in an increase when compared to neat epoxy. Minimum shear strength was obtained for 0.50 % Graphene added specimen as 12.32 ±1.97MPa. It can be inferred that Graphene`s increased the shear strength.

Nevertheless, the ultimate shear strain of the adhesives decreased by adding the nano particles into the epoxy resin. Maximum shear strain (at rupture) of Neat epoxy was obtained as 0.2 ±0.18 (mm/mm).The minimum rupture shear strain was obtained as 0.081 ±0.020 (mm/mm) for 0.25% Graphene added specimen and the maximum value of 0.15 ±0.11 (mm/mm) was obtained for 050Gr specimen.

Epoxy resin has the brittle behavior for lap shear tensile loading. After lap-shear tensile tests, the fractured surfaces were imaged by using SEM. Flat surfaces were emerged for the neat epoxy adhesive while the rough surfaces were imaged for hybrid adhesives. These results were compatible with the mechanical tests. Shear Modulus can be imagined as rigidity of a material. Shear modulus of neat epoxy adhesive was the highest as 101.6 MPa. Rigidity of the epoxy adhesive was variable by adding the nano particle in epoxy resin. Maximum shear modulus value was obtained for 1% Graphene added specimen as 120.75 MPa and the minimum was obtained as 51.12 MPa for 0.50 % Graphene added specimen.

The lap shear test is based on crack branching in nano modified epoxy based adhesives and SEM images showing progress compared to neat epoxy adhesive. SEM images obtained after tests of single-lap shear. The modified epoxy-based adhesives showed that the crack branches and progression increased when compared to neat epoxy. In addition, adding the graphene have been seen to prevent to crack propagation by bridging effect.

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