The Effect of MWCNT Addition on Mechanical Properties of Epoxy Based Adhesives

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Abstract – In this study, multi-walled carbon nanotubes (MWCNT) in different weight ratios effect on the mechanical properties of modified epoxy based adhesive under an axial load was investigated. 0.25, 0.50, 0.75 and 1 % by weight ratio the MWCNT added epoxy adhesives were prepared. Tensile test specimens of MWCNT added epoxy adhesives were produced in accordance with ASTM D638-10 and the tensile tests under axial load were performed. The tensile strength, elastic modulus, and toughness of the modified epoxy adhesives were calculated. As a result of these tests, tensile strengths and deformation of the samples were compared with reference (neat epoxy) samples and different proportions of MWCNT additive epoxy based adhesives. While the maximum strain of the epoxy adhesive is obtained as 0.061(mm/mm), it was obtained to 0.081(mm/mm) for 0.25% MWCNT added modified epoxy-based adhesive. In addition, fractured surfaces of the adhesives were analyzed by scanning electron microscopy (SEM) and damage mechanisms were investigated. By increasing the amount of MWCNT by weight, the agglomeration regions have been seen and because of these agglomerations, it has been observed to decrease in the mechanical properties such as tensile strength, etc.

Keywords – Tensile strength, SEM, Epoxy adhesives, MWCNT

I. INTRODUCTION

Because of the excellent physical and chemical properties, Epoxy resins become important adhesive among the current technologies and for the development of new technologies. Joints between the treated or untreated metal surfaces by using the adhesives made extensively properties instead of other joint techniques such as welding or soldering. For bonding similar or dissimilar materials, epoxy-based adhesives are extensively employed in many industries such as the aircraft industry[1, 2].

Adhesive bonding is used also in to medical applications[3]. The mechanical or physical property of the epoxy resin can be improved by adding the several nano particles[4]. 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[4]. 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[5]. 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[6].

II. MATERIALS AND METHOD

In this study, MGS-L285 lamination resin and MGS-H285 curing agent was used as adhesive and MWCNT as additives. 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. MWCNT were purchased from Nanografi Company. The diameter and the length of MWCNT used in this study have between 5 to 50 nm and 10 to 30 µm respectively.

A. Preparation of the Adhesives and Samples

Tensile specimens are produced in accordance with ASTM D638-10 standard. The dimensions of the drawing specimen produced according to ASTM D638-10 are shown in Figure 1.

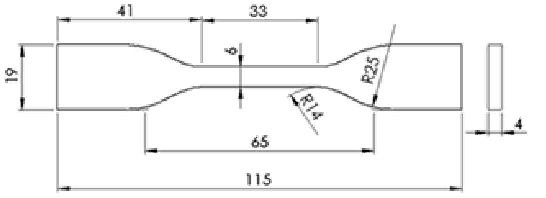

Figure 1. Tensile specimen according to ASTM D638-10.

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) MWCNT of determined amounts were added in to epoxy resin in a beaker.

(2) The epoxy resin and MWCNT 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 poured into sample mold.

(8) Samples were cured in an oven at room temperature for 24 h and then samples were kept for 15 hours at 80 °C for post-curing.

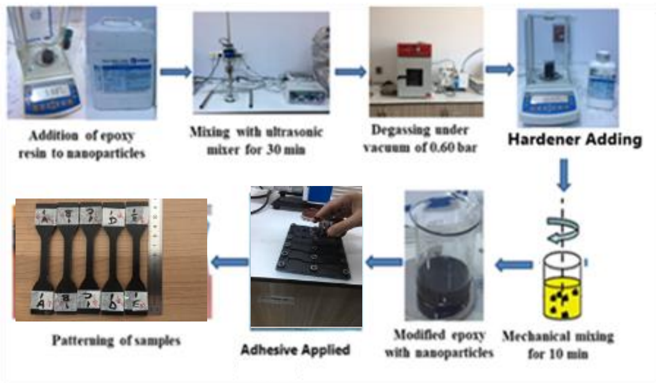

Fig. 2 Schematic flow chart of nanoadhesive preparation.

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

Table 1. Contents of the samples in wt %

Sample	**MWCNT**	**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

Tensile tests were carried out according to ASTM D638-10 standard. The schematic view of the sample was given in Figure 1.

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 applied region were measured by using Epsilon 3560 model biaxial extensometer.(see Figure 3).

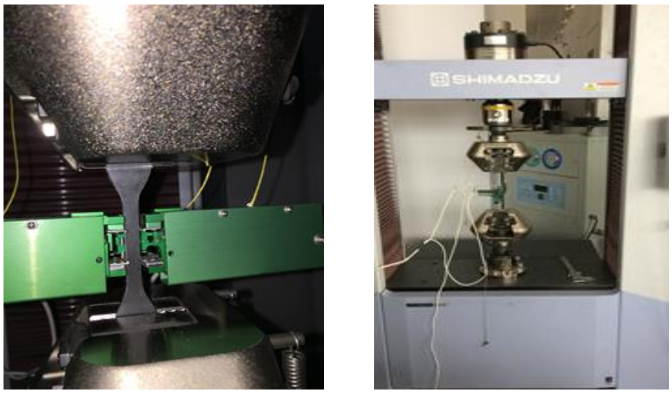

Figure 3. Tensile test at Shimadzu AGS-X test machine and using Epsilon 3560 extensometer

The tensile stress (σ) were calculated by the Equation 1.

$$\sigma = \frac{F}{A} \tag{1}$$

Where F is the applied force and A is force applied area. Engineering strain values were calculated by the Equation 2:

$$\varepsilon = \frac{\Delta L}{L} \tag{2}$$

Where the ΔL and L values are elongation and gage length of the tensile test specimens respectively.

D. Characterization

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

III. Results

A. Tensile Tests

In this study the 5(five) sets of the specimens were tested as seen Table 2. tensile test results are plotted in Figure 4 for various contents of the specimens.

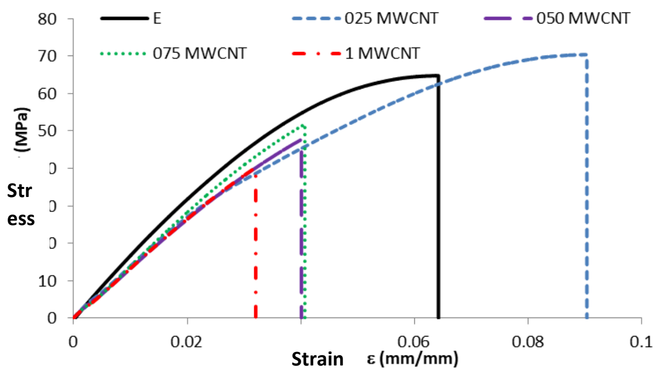

Figure 4. Tensile test results for 0.25, 0.50, 0.75 and 1 wt% MWCNT and Neat Epoxy.

Table 2. Mechanical Properties of samples

Sample	Tensile Strength (MPa)	Tensile Strain (mm/mm)
Neat	58.7 ±8.63	0.061 ±0.007
025CNT	66.48 ±7.59	0.081 ±0.020
050CNT	42.32 ±5.97	0.051 ±0.08
075CNT	45.38 ±8.61	0.045 ±0.063
1CNT	37.7 ±6.26	0.024 ±0.048

B. SEM Images of the Fractured Surfaces

SEM images of the fractured surfaces of Neat, 025CNT, 050CNT, 075CNT and 1CNT samples after the tensile tests of specimens were presented in Figure 5a, b, c, d, and e respectively at 10kX magnification. As seen in Figure 5a, the fractured surface of the neat epoxy sample is flat and smooth. But by adding the MWCNT into epoxy, the fractured surfaces become roughed and crack progressions were observed to be inhibited. It can be seen in Figure 7b that MWCNT were restricted to crack progression by bridging and pull out systems. This effect can be seen for lower ratios of MWCNT as given in Table 2. Tensile strengths of the less additive contented samples are smaller than to neat epoxy specimen except for the 025CNT. Also, the maximum strain value was reached for the 025CNT sample. The mixtures were tried to prepare homogeneously. However, there are agglomerated regions in Figure 5e by increasing the amount of MWCNT. Because of the inhomogeneity for higher additive ratios of the mixtures, the decreasing of the strength can be mentioned.

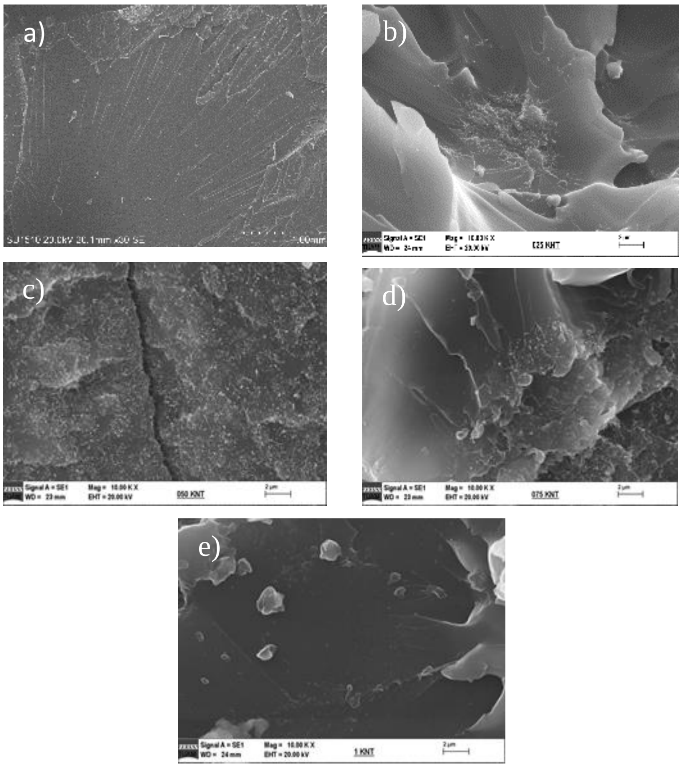

Figure 5. SEM Images of fractured surfaces of a) Neat b) 025CNT c) 050CNT d) 075CNT e) 1CNT samples after tensile tests

IV. DISCUSSION

In this study, epoxy based nano adhesives were produced and obtained their mechanical properties. For obtaining the mechanical properties of the adhesive the tensile specimens were produced and performed to tensile tests. The maximum tensile strength was reached for the 025CNT specimen while the minimum value was obtained for the 1CNT specimen.

V. CONCLUSION

In this study, the tensile strength of the epoxy-based hybrid adhesives was investigated. MWCNT was added into epoxy resin to examine the mechanical properties of epoxy. Nanoparticle (MWCNT) ratios (in % weight) were selected as 0.25, 0.50, 0.75 and 1. Results were compared with the neat epoxy adhesive. First, the effects of MWCNT on to tensile strength of the epoxy adhesive were observed. Later SEM images of the ruptured samples' surfaces were evaluated.

In order to obtain the tensile strength of the epoxy adhesive, Tensile Tests were performed. while the maximum tensile strength was 66.48 ±7.59 MPa for 0.25% MWCNT added specimen, Tensile strength of Neat Epoxy was obtained as 58.77 ±8.63 MPa. The tensile strength of the produced epoxy-based adhesive by using MWCNT resulted in an increase only for 025CNT specimen when compared to other adding ratios or neat epoxy. Minimum tensile strength was obtained for 1 % MWCNT added specimen as 37.7 ±6.26 MPa. It can be inferred that the tensile strength decreases for increasing rates except for 025CNT. Likewise, the ultimate tensile strain of the adhesives decreased by adding the nanoparticles into the epoxy resin. The maximum tensile strain of Neat epoxy was obtained as 0.061 ±0.007

(mm/mm). The minimum tensile strain was obtained as 0.024 ±0.048 (mm/mm) for 1% MWCNT added specimen and the maximum value of 0.081 ±0.020 (mm/mm) was obtained for the 025CNT specimen.

Cured epoxy has brittle behaviour for tensile loading. After tensile tests, the fractured surfaces were imaged by using SEM. Flat surfaces emerged for the neat epoxy adhesive while the rough surfaces were imaged for hybrid adhesives. These results are compatible with mechanical tests. As a result of investigations, it can be said that adding MWCNT at 0.25% weight ratio into epoxy resins increases the tensile strength and maximum strain of epoxy adhesives. These property enhancements can be concluded that the crack propagations inhibited by bridging and pull out effects.

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