European Journal of Technique

journal homepage: https://dergipark.org.tr/en/pub/ejt

Vol.11, No.2, 2021

Investigation of the Effects of Use of GNP and GNP Reinforced Nano-Fibers with Epoxy Adhesive on Tension Tests

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ARTICLE INFO

Received: Jun., 25. 2021
Revised: Oct., 04. 2021
Accepted: Oct, 13. 2021

Keywords:
Adhesive
GNP
Hybrid joining
Nano-Fiber

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ISSN: *2536-5010* | e-ISSN: *2536-5134*

DOI: https://doi.org/10.36222/ejt.957654

ABSTRACT

In this study, the effects of different ratios graphene nanoparticles (GNP) and Nanofibers were investigated on bonding joints. For this, AA5754 alloy samples were bonded by DP460 adhesive to different ratios of graphene nanoparticle (GNP) and Nanofibers. Reinforcement ratios were chosen as 0.1% wt., 0.2% wt., and 0.3% wt. Adherent samples of 75x30x3 mm and patches of 40x30 mm were used, which cut from AA5754 alloy plate. Tensile tests of samples were conducted to see the effects of each parameter on the bonding joints after the bonding process. After tension tests, macrostructure and SEM images of the rupture surfaces were taken. Tensile test results, the best tensile damage load has been obtained in bonding joints where Nanofiber has been used together with 0.2% wt GNP. In addition, it has been observed that CHZ + Fiber + CHZ and AHZ + CHZ structures seen in macrostructure images have positive effects on tensile test results.

1. INTRODUCTION

The method of joining by the adhesive is widely used because it offers significant advantages in many technological fields such as aviation, construction, automotive, and marine. However, there may be many problems due to some disadvantages as well as the advantages obtained in adhesive bonding methods. One of these problems is the occurrence of stress intensities at the edges of adhesive joints. There are different methods to reduce these stresses, such as thinned the bonding tip, created an adhesive radius, changing the overlap geometry, and hybrid double overlap [1-9]. Marannano et al. [5] were carried out a studying to experimentally and numerically test the mechanical behavior of hybrid bonded/riveted joints. As a result of the experiments, static and fatigue values were respectively obtained 20% and 45% higher in the hybrid joints type created using rivet in bonding joints. Various techniques have been tried to establish a hybrid joining type in adhesive bonded joints and obtain a more robust structure at the same time. One of them is the joining made by adding nano-particles into the epoxy adhesive. Gültekin et al. examined the impacts of nano-graphene powder reinforcement on epoxy bonding bonds. According to their results, nano-graphene reinforcement has a significant effect on adhesive joints [10]. Akpinar et al. investigated the effects of 0.25% wt., 0.5% wt., 1% wt., 2% wt., and 3% wt. Nano-graphene-COOH, Carbon Nanotube-COOH, and Fullerene C60 supplements on single lap bonded joints. They stated that the reinforcements used in different rates have different effects on the adhesive bonded joints based on the mixing ratio and the type of reinforcement. In addition, they were indicated the best recuperation was obtained from 1% wt. of Fullerene C60 supplement [11]. Jia et al. tried to determine the effect of graphene nanoplatelets-reinforced adhesive on the Mode I fracture resistance in double cantilever beam joints. They stated that 0.25% wt. graphene nanoplatelets reinforcement has a strength of 5 times higher than the pure epoxy adhesive on the breaking resistance of the joints. They also have emphasized that as the graphene nanoplatelets ratio increased, the toughness of the joints decreased [12]. Moriche et al. endeavored to determine the impacts of GNP/epoxy nanocomposite adhesives on thermal conductivity and shear resistance. They stated that thermal conductivity increased by 206% and 306% respectively as a result of adding 8% and 10% graphene nanoplatelets into the epoxy adhesive. They also expressed that there was no clear effect on shear resistance [13]. Khoramishad et al. investigated the effect of temperature on graphene oxide nano-platelet reinforced nanocomposite adhesive joints. They stated at the end of the experiments performed on 1% wt. and 3% wt. graphene oxide nano-platelet supplements that the critical temperature of the 1% reinforcement ratio was 60 °C and the critical temperature of 3% wt. reinforcement ratio was 40°C [14]. Sadigh and Marami investigated the tensile and compressive stresses of the joints made at different extension ratios with reduced graphene oxide (RGO) reinforcement into the epoxy adhesive, experimentally and numerically. In the joints made with 0.5% reduced graphene oxide reinforcement, the tensile and compressive

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stresses were 30% and 26% better, respectively, compared to the pure epoxy connections. They stated that successful results were obtained when the experimental and numerical results were compared. [15].

When the literature studies were examined, many studies have been conducted on the use of adhesive materials with various nanoparticles. However, it has not been seen that Nano-Fiber as an interlayer has been used in any study. In this study, Nano-Fibers produced with 1% wt. GNP reinforcement by Electro-spinning method were used as an intermediate layer in order to investigate this situation. in addition, adhesions were made with DP460 adhesive and an adhesive mixture prepared with 0.1% wt., 0.2% wt., and 0.3% wt. GNP reinforcement to this adhesive. After the bonding process, the samples were subjected to tensile tests in order to see the effects of each parameter on the bonding strength. Finally, after the tensile tests, the macrostructure and microstructure examinations of the separated surfaces were made.

2. EXPERIMENTAL PROCESSES

In this study, the adherent material was used the AA5754 aluminum alloy, which provides spectacular performance in extreme areas of use. It has a high resistance to seawater and industrial chemicals. AA5754 aluminum alloy has a high weldability and fatigue strength; it resists seawater corrosion and has good cold workability in soft temper form. Because of these properties, it is widely used in the shipbuilding industry, chemical devices, storage tanks, pressure vessels, folding bridges, welded truck bodies, military vehicle bodies, and armors [16]. In Figure 1, it was given the stress-strain graphic of AA5754 alloy [17]. DP460 was utilized as the adhesive material. This material has two components as epoxy and intensifier electrode (accelerator). The adhesive displays high resistance when the epoxy/accelerator ratio is 2/1. Special holozoic ends were used to provide this ratio. Figure 2 shows the stress-strain graphic of DP460 adhesive material. In this study, which used the adhesive bonding technique, Nano graphene particle reinforced nano-fibers produced by electro-spinning method and Nano Graphene particles (GNP) adding to DP460 adhesive were used to obtain a better bond strength in bonding joints. Nano graphene particle thickness is 5-8 nm; diameter was 5 μm; the surface area is 120-150 m2/g; purity was 99% and density is 0.05 g/cm3. Figure 3 shows the macro and micro images of graphene used in the experimental study.

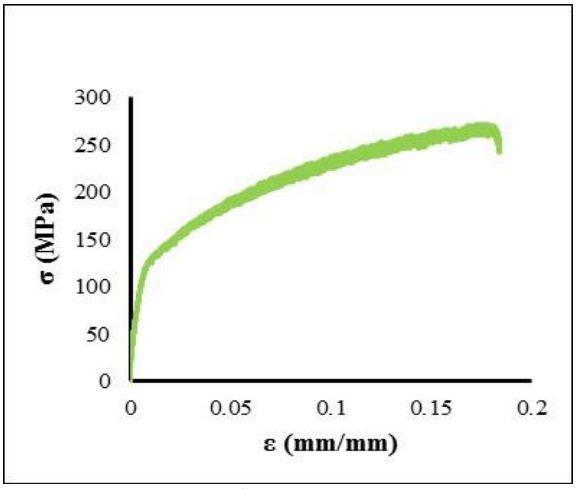

Figure 1. Stress-strain graphic of AA5754 material

TABLE 1
AA5754 MECHANIC PROPERTIES

Elastic Modulus	78.586GPa
Slip Modulus	25.9GPa
Poisson's Ratio	0.324
Yield Strength	140 MPa
Tensile Strength	272.0561MPa

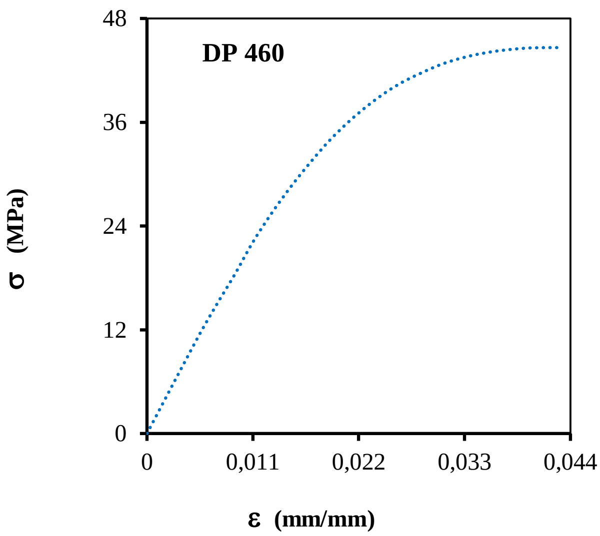

Figure 2. Stress-strain graphic of DP460 material

TABLE 2
DP460 MECHANIC PROPERTIES

Elastic Modulus	2077.1 MPa
Poisson's Ratio	0.38
Tensile Strength	44.616 MPa

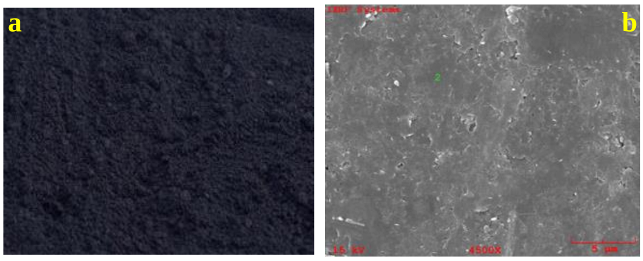

Figure 3. Macro and SEM images of the graphene particle used in the experimental study (a-Macro; b-Micro)

2.1. Preparing double patched test samples

First of all, in this study, the AA5754 aluminum plate of 3 mm thick, 2000 mm long, and 1000 mm wide was provided for the bonding process. The aluminum plate in 2000x1000x3 mm dimensions was cut into samples of 30 mm width and 75 mm length for experimental study. Similarly, the parts that were utilized as patches in the bonding process were cut from the AA5754 aluminum alloy plate in 40x30x3 mm dimensions. In the final processings of the experiment samples, the preparation of the region where the bonding process could be performed was. For this, the regions to be contacted with the adhesive were cleaned with 100-sized sandpaper. After the cleaning process, it was kept in acetone for 10 minutes. After acetone treatment, samples and patches were cleaned with water and left to dry. In Figure 4, an image of the sample and patches used in the bonding process is given.

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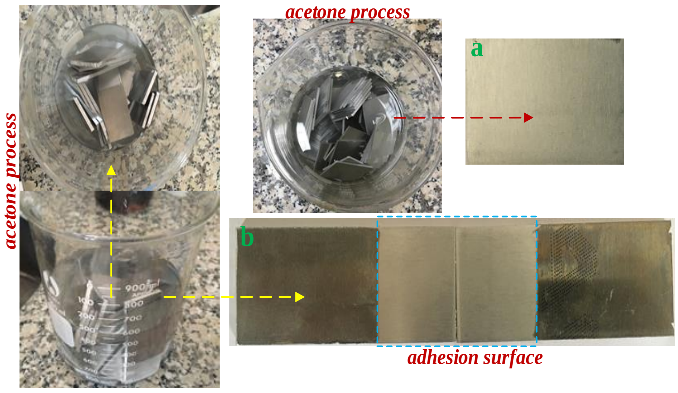

Figure 4. Preparation of sample and patch used in bonding (a- Patch sample b- Adherent sample)

2.2. Preparation of neat and graphene reinforced adhesives

The bonding process was started after the process of preparation of samples. Firstly, the bonding process was done using DP460 adhesive. Figure 5, shows DP460 adhesive and the equipment used in bonding with graphene reinforced. All these types of equipments were obtained from 3M Company. After the bonding process using DP460 adhesive, nano-sized graphene particles were added to the DP460 adhesive at the rates of 0.1% wt., 0.2% wt. and 0.3% wt., and bonding was carried out. A homogeneous mixture was obtained by using an ultrasonic mixer to obtain a good adhesion from graphene particles added in different proportions in DP460 adhesive. It was mixed DP460 adhesive and nano-sized graphene particles in an ultrasonic mixer for 15 minutes and 30 Hz at room temperature conditions for this process (Figure 6).

Figure 5. Equipment used in the bonding processing

2.3. Preparing nanofibers with graphene reinforced

1% Graphene reinforced nanofibers were used as an intermediate layer between DP460 adhesive and AA5754 aluminum sample and the same material-based patch piece in the experimental study. The electro-spinning method was used for the production of graphene reinforced nanofibers used in this study. The electro-spinning test device consists of three parts. Related parts are the polymer solution supply system, high voltage power supply, and cylindrical collector (Figure 7).

Commercially available Polyvinyl butyral (PVB) polymer, hardener Tetraethyl orthosilicate (TEOS), and ethanol were used to produce nanofibers in the electro-spinning method. Molecular weight and melting temperature vary between 165-185°C in PVB 40000-70000 g/mol. These properties are important. Because the solution, which significantly affects the structural and morphological properties of nanofibers, must be prepared based on these properties.

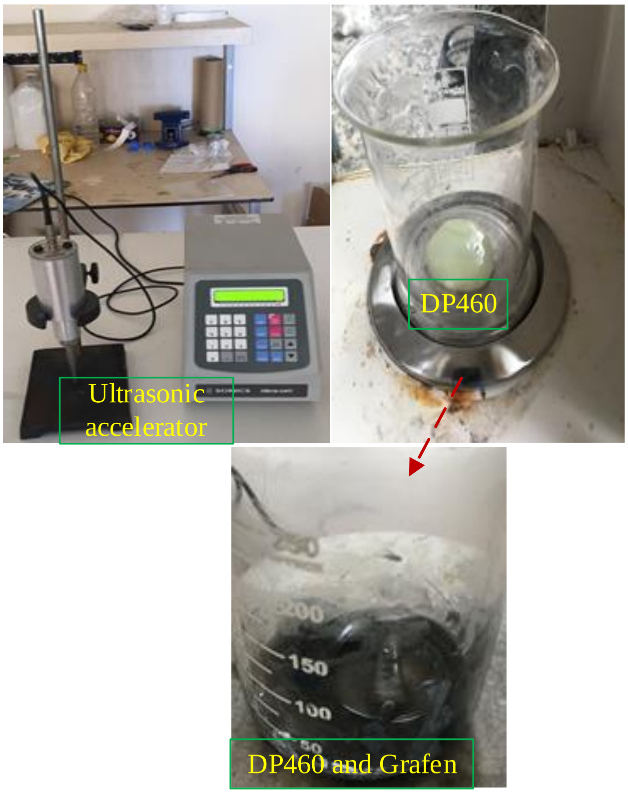

Figure 6. The ultrasonic mixing process of DP460 and Graphene

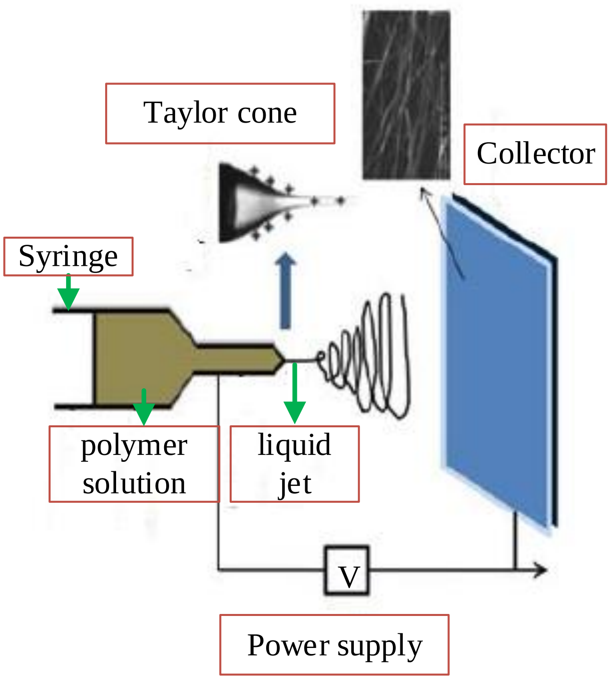

Figure 7. Electro-spin Device Schematic View [17]

PVB polymer was mixed with a magnetic stirrer for 4 hours at 50 ^{0}C in ethanol with a ratio of 10% by weight [18, 19] in the solution preparation process. In this mixing process, 50% TEOS was added according to the amount of polymer and the mixture was mixed until it became homogeneous and stable. Graphene nanoparticles were mixed in ethanol for 10 minutes at low frequencies (15 kHz) with the help of an ultrasonic probe to be homogeneous. Then, solutions were prepared by adding PVB polymer and TEOS so that the GNP ratio in the mixture was 1% wt. It was transferred into a sterile syringe and placed on the automatic syringe pump after the solution was prepared. The distance between the collector and the metallic needle tip was arranged so as to be 15 cm. It was chosen a cylindrical mandrel as a collector and aluminum foil was wrapped on it to collect nanofibers in terms of ensuring homogeneity. The speed of the cylindrical mandrel was 400 rpm while nanofibers were collected. The prepared nanofibers were left to dry in the drying oven at 40 ^{0}C for 30 minutes. Figure 8 shows the processes of nanofiber production; Figure 9 shows produced nanofiber.

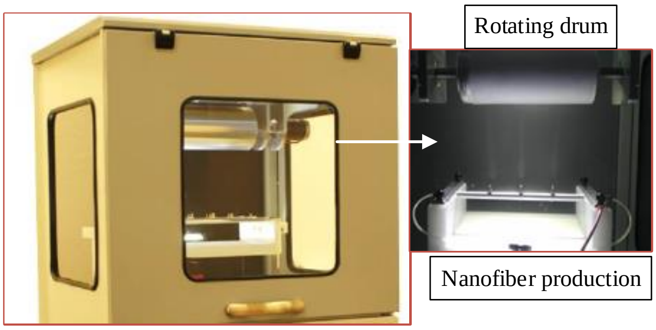

Figure 8. Schematic picture showing the electrospinning process; the image of Nano-Fibers produced with a multi-needle tip

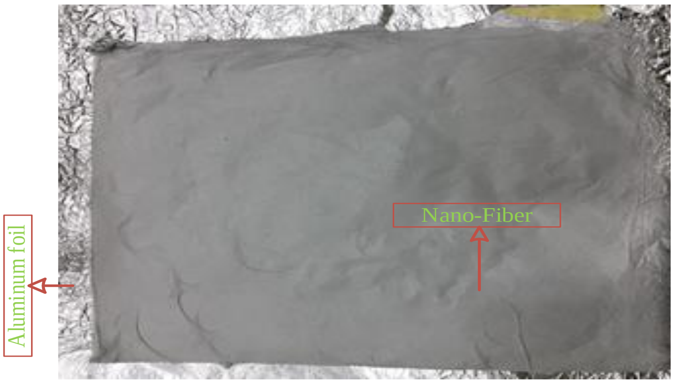

Figure 9. Nano-fiber, which particle reinforced

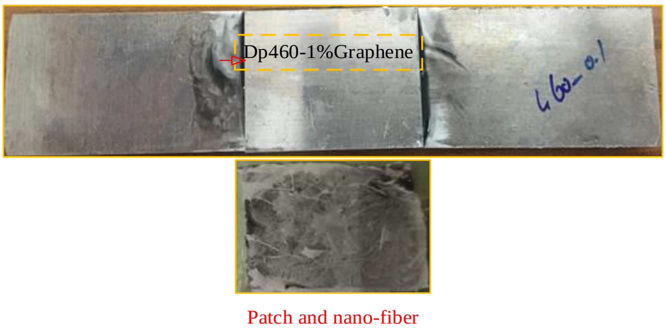

Figure 10. Nano-fiber and 1% wt. GNP reinforced bonding process

2.4 Bonding processing and tensile tests

It was performed tensile tests after the specimens were carried out bonding process (Figure 10). Tensile tests were carried out in SHIMADZU AG-IC brand tensile tester with 250 KN capacity at a constant feed rate of 1mm/min (Figure 11). In the tensile test repeated 3 times for each parameter, the average tensile force was determined and the margin of error was minimized. Then, force-elongation curves were obtained by using these values.

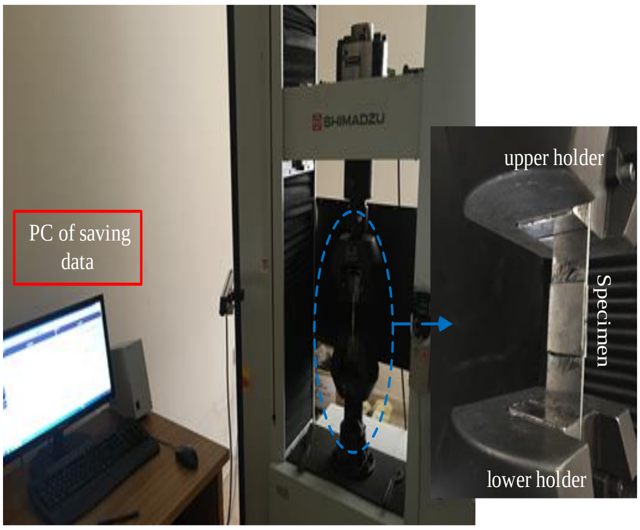

Figure 11. Tensile experiment processes

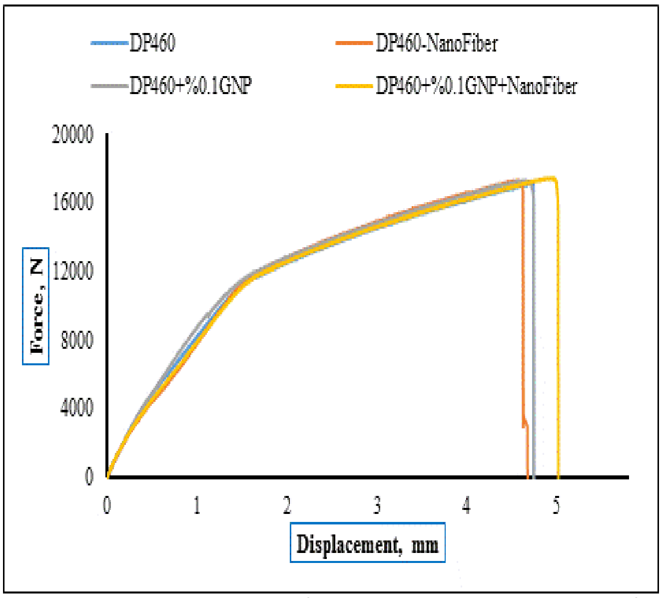

Figure 12. Force and elongation of the joints using 0.1% GNP and Nanofiber

3. RESULTS AND DISCUSSION

In this study, AA5754 aluminum alloys were bonded with double patches. For the bonding process, DP460 adhesive and together with this adhesive, 0.1% wt., 0.2% wt., 0.3% wt. Graphene nanoparticles and 1% Graphene nanoparticle reinforced Nano-Fibers were used. After the bonding processes, the effect of each parameter on the bonding tensile force was investigated. For this, the tensile tests of the samples bonding in each parameter were made. After the tensile tests were completed, their structures on the adhesion surface were examined. Therefore, macro and micro images of the adhesion region were taken after the tensile test of each sample. Figure

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12, Figure 13, and Figure 14 were given force-elongation graphics of the samples using Graphene nanoparticles and Nanofibers produced with 1% wt. Graphene nanoparticle reinforcement.

3.1 Tensile Tests

Tensile test results of bonding joints using DP460 adhesive, GNP, and Nanofibers are given in Figure 12, Figure 13 and Figure 14.

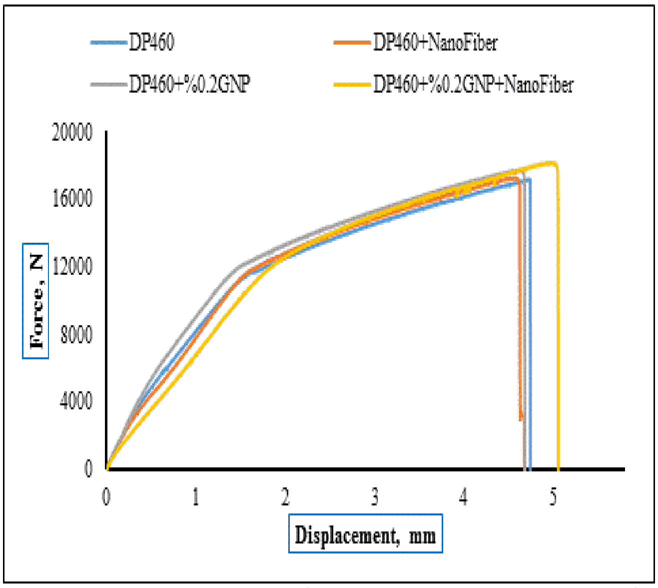

Figure 13. Force and elongation of the joints using 0.2% GNP and Nanofiber

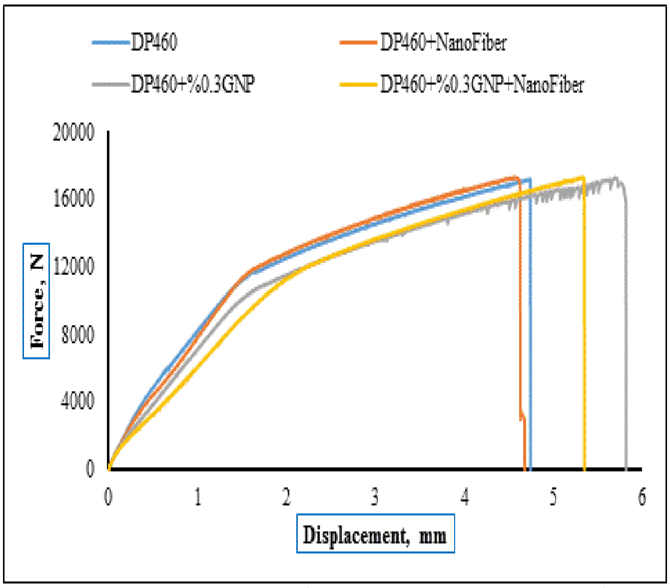

Figure 14. Force and elongation of joints using 0.3% GNP and Nanofiber

In Figure 12, the force-elongation graph of double-patch AA5754 aluminum alloy samples using DP460 adhesive and 0.1% wt. graphene nanoparticles added to this adhesive and nanofibers produced with 1% graphene nanoparticle addition is given. Considering the force-elongation values obtained by adding graphene nanoparticles and nanofiber to DP460 adhesive in Figure 12. It is seen that the using of 0.1% graphene nanoparticles and nanofibers did not make a significant change on the adhesive joints, but higher tensile damage force and elongation were obtained in the joints where 0.1% wt. graphene nanoparticles and nanofibers were used together. The best adhesive-bonded joint was obtained from the joint using 0.2% GNP and Nano-Fiber together in bonding joints obtained with 0.2% GNP and Nano-Fiber reinforcement. Similarly, as is in Figure 12, a higher tensile damage load was obtained from adhesive bonded joints where 0.2% GNP and Nano-Fiber were separately used compared to joints using DP460 adhesive. We can see a partial improvement when looking at the force-elongation values in Figure 13. Also, it clearly, it can states that the reinforcement ratio has a significant effect here. Saraç et al. In their study stated that nanoparticle reinforcement ratios and types have a significant effect on adhesive joints [20].

The joints obtained with 0.3% wt. GNP reinforcement (Figure 14) showed a different behavior than the bonding joints using 0.1% wt. and 0.2% wt. GNP and nanofibers. Here, the highest tensile force and elongation were obtained from the joints obtained with the reinforcement of 0.3% wt. graphene nanoparticle. Similarly, Topkaya et al. stated that GNP reinforcement has a positive effect on the bonding tensile force of the joints. They also emphasized the importance of 0.2% wt. GNP supplementation in their study [21].

The better tensile damage force was obtained than the bonds obtained with DP460 adhesive in all bonding joints where 0.1% wt., 0.2% wt. and 0.3% wt. graphene nanoparticles and nanofibers produced with 1% graphene nanoparticle reinforcement were used. However, the best tensile damage force was obtained from adhesion joints where nanofiber and GNPs were used together. In addition, the best tensile damage force was obtained as 18183.8 N from the joints used nanofibers produced with 0.2% graphene nanoparticle and 1% graphene nanoparticle reinforcement. When the force-elongation graphs are examined, the elastic behavior of the bonded joints using 0.1% GNP and nanofiber is linear. However, there is a partial fluctuation in the elastic region in the connections where 0.2% GNP, 0.3% GNP and nanofiber are used. There was also an increase in yield force relative to 0.1% GNP and nanofiber. Table 3 gives values for all tensile damage forces.

TABLE 3
RESULTS OF TENSILE TESTS

Reinforcement (%)	Force (N)
DP460	17187.5
DP460 + Nanofiber	17312
DP460+0.1Graphane	17281.3
DP460+0.1Graphane+NanoFiber	17453
DP460+0.2Graphane	17730.5
DP460+0.2Graphane+NanoFiber	18183.8
DP460+0.3Graphane	17265.6
DP460+0.3Graphane+NanoFiber	17289.1

3.2. Macro and micro images of surfaces of adherence

Obtaining a good joining structure in the joints made with adhesive depends on a good surface of adherence between the samples bonded with the adhesive. Therefore, it is essential to clean the surface of adherence of the samples bonded in the joints made with the adhesive. It needs to review the surface morphological structure of the joints to observe the effects of the processes and adhesion technique on the adhesion quality. In this study, photographs of the ruptured surface regions of each sample were taken after the tensile tests to observe the structural effects of nanoparticles and nanofibers on tensile damage loads. It was tried to determine adhesion mechanisms on the surface of the bonding of samples. Finally, the relation or effects of mechanisms between the adhesion damage loads were explained. In Figure 15, firstly was given the Nano-fiber SEM images.

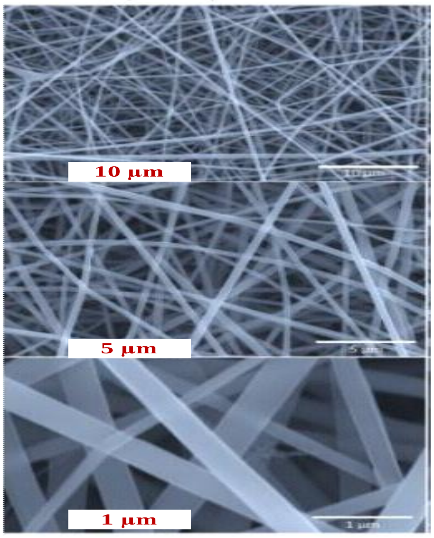

Figure 15. SEM images of Nanofiber

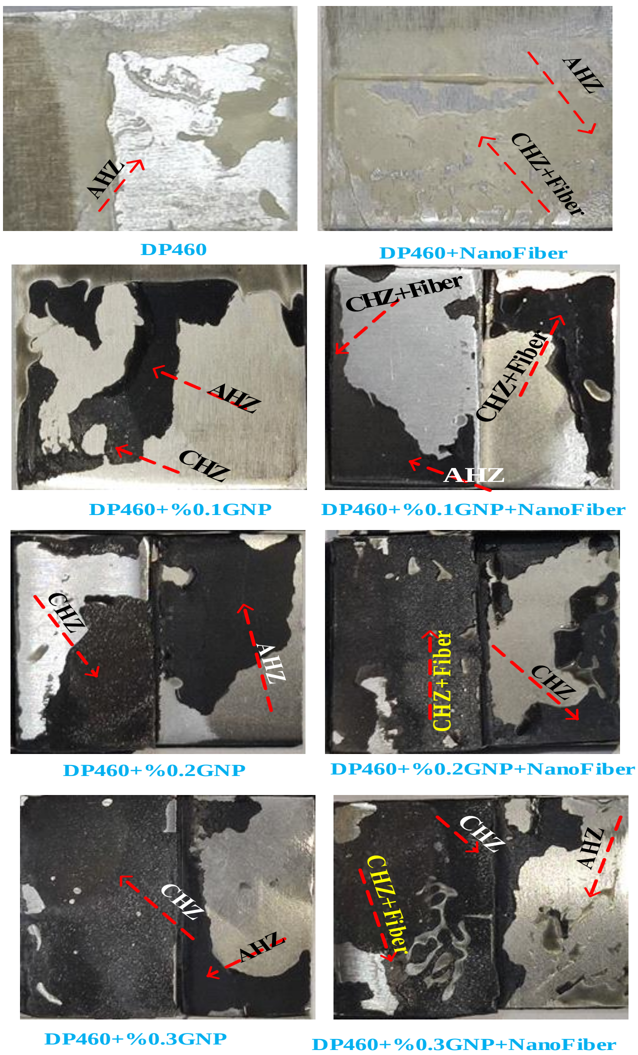

Figure 16. Surfaces of adherence of samples separated by tensile tests

In Figure 15, SEM images of the morphologies of different sizes of PVB / TEOS / 1GNP nanofibers produced by electro-spinning are given. Examination of the SEM images, it is understood that the nanofibers have a homogeneous structure.

Macro and SEM photographs of the ruptured surface of adherence of each sample were given in Figure 16 and Figure 17 after the tensile tests.

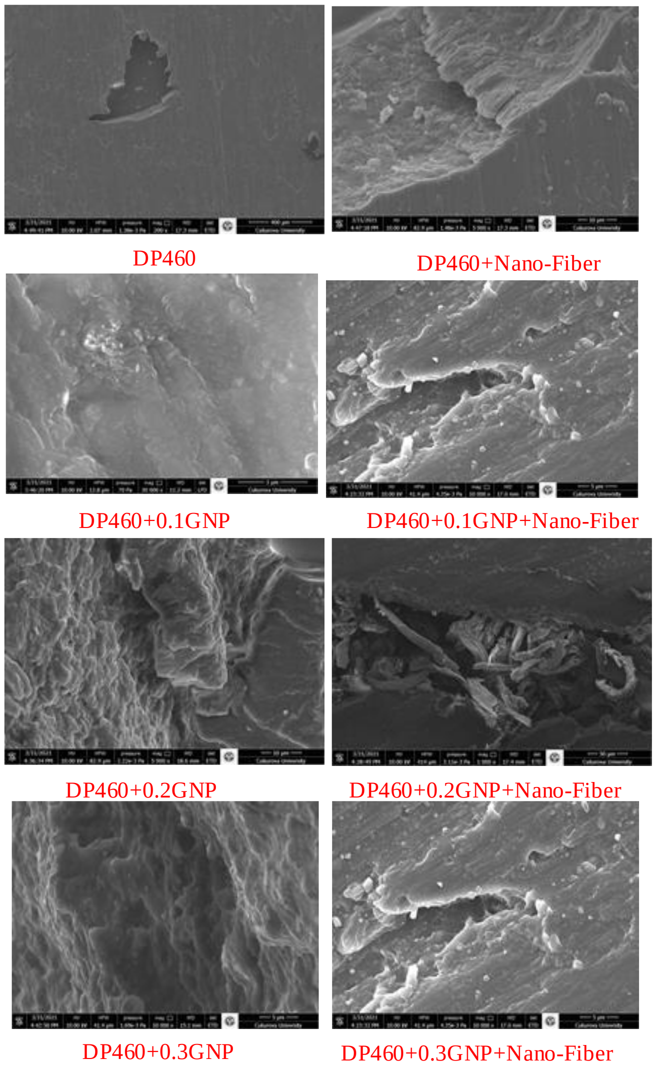

Figure 17. SEM images of the surfaces of samples separated by tensile tests (10000X-5µm)

Only adhesion (AHZ) was seen in joints DP460. Adhesion (AHZ) and cohesion (CHZ) were observed together in the joints where NanoFibers with Graphene nanoparticle and Graphene nanoparticle reinforcement were used. There was also made a definition as CHZ+Fiber in joints with nanofibers. It is seen when tensile test results were compared with surfaces of CHZ + Fiber, CHZ, and AHZ structures that there are better results in surfaces with CHZ+Fiber and CHZ. It is thought that there are enhancing effects on tensile forces on surfaces of CHZ+Fiber +CHZ and AHZ+CHZ structures (figure16). When SEM images were examined (figure17), it was seen that there was a thin and long microstructure on the rupture surface of the bonding made with DP460 + 0.2GNP + Nano-Fiber parameters. It is thought that this microstructure is formed by the effect of nanofibers used as reinforcement elements. In addition, the reason why the joint made with this parameter rupture at maximum load is attributed to the transport of the load on these homogeneous thin long fibers.

4. CONCLUSION

AA5754 aluminum alloys were bonded with double patches using DP460 adhesive and also used adhesive mixtures prepared with 0.1% wt., 0.2% wt., and 0.3% wt. GNP added to these adhesives. Nanofibers produced with 1% GNP reinforcement were also used with DP460 adhesive and the adhesive mixtures of GNP. Subsequently, samples bonded were subjected to the tensile test to determine the effect of Nano-fiber and GNP reinforcement ratios on bonding joints. Finally, the adhesion mechanisms of each sample were determined by taking macro images and SEM of the fracture surfaces after the tensile process was completed. All results obtained were provided below.

✓ Tensile forces of double patched joints made with a mixture of DP460 adhesive and Graphene nanoparticle (GNP) were obtained as higher than tensile forces of double-patch joints achieved using only DP460 adhesive.

✓ The tensile forces partially increased in double patched joints where nanofibers were used together with DP460 adhesive.

✓ The tensile forces of the joints including adhesive prepared with the reinforcement of nanofibers and graphene nanoparticle (GNP) to DP460 adhesive provided the highest values. The best tensile force was obtained as 18183.8 N from samples where 0.2% graphene nanoparticles and Nano-Fibers were utilized together.

✓ AHZ, CHZ + Fiber + CHZ, and AHZ + CHZ surface structures were formed on the surfaces of bonding. In addition to all these, good tensile damage loads were obtained in samples with CHZ + Fiber + CHZ and AHZ + CHZ surface structures.

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BIOGRAPHIES

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