FEBRUARY 2021 M.Sc. in Mechanical Engineering HARUN KARAOĞLAN


REPUBLIC OF TURKEY

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# INVESTIGATION OF THE EFFECTS OF NANOPARTICLES ON ADHESION PROPERTIES OF SINGLE STRAP REPAIR IN COMPOSITES

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HARUN KARAOĞLAN


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**Supervisor**

**Prof. Dr. Ahmet ERKLİĞ**

**By**

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**I hereby declare that all information in this document has been obtained and presented in accordance with academic rules and ethical conduct. I also declare that, as required by these rules and conduct, I have fully cited and referenced all material and results that are not original to this work.**

**Harun KARAOĞLAN**

## ABSTRACT

**INVESTIGATION OF THE EFFECT OF NANOPARTICLES ON ADHESIVE OF SINGLE STRAP REPAIR IN COMPOSITES**

**KARAOĞLAN, Harun**
**M.Sc.in Mechanical Engineering**
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Composite materials have been thoroughly using in engineering application areas such as electronics, aerospace, automotive. Due to the production of new fibers such as carbon, boron, and aramids, and new composite structures of matrices made of metals and ceramics since the 1970s, the use of composites has been growing thoroughly. In engineering applications areas, adhesively bonded repairs are expanding mechanical repair alternatives and various nano materials are added to increase the strength of the bonding surface of adhesive bonded repairs. In this thesis, the effects of tensile features of epoxy adhesive with nanomaterials (nano-silica, nano-graphene, and nano-clay) on the glass epoxy composite laminates adhesively bonded single strap repairs are investigated. Nano-silica, nanographene, and nano-clay particles were added to the epoxy resin with an amount of (1, 2, 3, 5, and 10 wt. %), (0.05, 0.1, 1, 3, and 5 wt. %), and (1,2,3,4, and 5 wt. %) respectively. Single strap repairs were used as different patch ratio (D/d=2, and D/d= 3). 10 mm diameter patch holes were opened by a CNC machine. The samples were subjected to tensile testing and their load carrying capability was measured. Experimental results showed that a 3 wt. % ratio of nano-clay particles have the best tensile force values of all nanoparticles in the epoxy resin.

**Keywords:** Adhesively Bonded, Nano-silica, Nano-graphene, Nano-clay, Single-strap Repairs, Tensile Test.

# ÖZET

## TEK ŞERİTLİ ONARIMLARDA YAPIŞTIRMA BAĞLANTILARINDA NANO PARÇACIKLARIN ETKİSİNİN ARAŞTIRILMASI

**KARAOĞLAN, Harun**
**Yüksek Lisans, Makine Mühendisliği**
**Danışman: Prof. Dr. Ahmet ERKLİĞ**
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Kompozit malzemeler elektronik, havacılık, otomotiv gibi mühendislik uygulama alanlarında kapsamlı bir şekilde kullanılmaktadır. 1970'lerden beri karbon, bor ve aramidler gibi yeni elyafların üretimi ve metal ve seramikten yapılmış matrislerin yeni kompozit yapıları nedeniyle, kompozitlerin kullanımı iyice artmaktadır. Mühendislik uygulamaları alanlarında, yapışkanla bağlanan onarımlar genişleyen mekanik onarım alternatifleridir ve yapışkanla bağlanan onarımların yapıştırma yüzeyinin mukavemetini artırmak için çeşitli nano malzemeler eklenir. Bu tez çalışmasında, cam epoksi kompozit laminatların tek şerit onarımlarında epoksi yapıştırıcının nano malzemeler ile (nano silika, nano grafen ve nano kil) güçlendirilmesinin çekme özelliklerine etkileri araştırılmıştır. Nano silika, nano grafen ve nano kil parçacıkları epoksi reçineye sırasıyla ağırlıkça (%1, 2, 3, 5 ve 10), (%0,05, 0,1, 1, 3 ve 5), ve (%1, 2, 3, 4 ve 5) oranlarında katılmıştır. Tek şerit onarımları farklı yama oranlarına (D / d = 2 ve D / d = 3) göre gerçekleştirdi. Yama delikleri CNC ile 10 mm çapında açılmıştır. Numuneler çekme testine tabi tutuldu yük taşıma kabiliyetleri ölçüldü. Deneysel sonuçlar, nanokil parçacıklarının ağırlıkça %3'ünün epoksi reçinesindeki tüm nano parçacıklar arasında en iyi çekme kuvveti değerlerine sahip olduğunu göstermiştir.

**Anahtar Kelimeler:** Yapışkanla Bağlanmış, Nano-silika, Nano-grafen, Nano-kil, Tek-Şeritli Onarımlar, Gerilme Testi.

**for my family and grandfather…**

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## LIST OF ABBREVIATIONS

| | |
|---|---|
| **CMCs** | Ceramic Matrix Composites |
| **MMCs** | Metal Matrix Composites |
| **PMCs** | Polymer Matrix Composites |
| **C/C** | Carbon/Carbon |
| **SiC** | Silicon Carbide |
| **MWCNTs** | Multiwall Carbon Nanotubes |
| **Wt** | Weight |
| **CNPs** | Clay Nano Particules |
| **PVA** | Polyvinyl acetate |
| **SNPs** | Silica Nano Particules |
| **GNPs** | Graphene Nano Particules |
| **SSA** | Sewage Sludge Ash |
| **GONPs** | Graphene Oxide Nano-Platelets |
| **SGFR** | S-Glass Fiber Reinforced |
| **SiO2** | Silicon Dioxide |
| **GPa** | Giga Pascal |
| **D/d** | Patch Diameter(D) and Hole Diameter(d) |
| **SSR** | Single Strap Repair |
| **Tg** | Glass Transition Temperature |

# CHAPTER 1

# INTRODUCTION

Composite materials are increasingly used in industry due to composite materials are used extensively in engineering application areas. One of these usage areas is the adhesively bonded repair technique. These advantages are lower edge stress concentration factors, more uniform distribution of stress, lighter weight, lower fabrication cost, etc.

## 1.1 Adhesive Bonding

In engineering applications, adhesively bonded joints are increasing as an alternative to mechanical fastening which has many benefits over traditional mechanical seals. Adhesive bonding gives a structural design opportunity that cannot be accomplished with any other technique. In comparison, adhesively bonded systems are distinguished by lower weight in the majority of load cases (tension, compression, shear). Gas and moisture tight (unlike riveted or bolted joints) are fused joints with good toughness, great corrosion resistance, and good fatigue properties. At present, structural bonding is still not commonly known alternating to riveting. However, the positives of adhesive bonding make up for the drawbacks if any disadvantages are overcome. The current problem is therefore to overcome drawbacks such as the electrical insulating properties, water sorption, and then debonding flammability, and critical points interested in the processing temperature [1].

Adhesive bonding is a method of substance bonding in which an adhesive applied between the surfaces of the adhesive solidifies to form an adhesive connection. In engineering applications areas, adhesively bonded joints are expanding mechanical joints alternatives and provide several advantages over traditional mechanical connectors. Lower structural weight, lower production costs, and increased resistance to loss are among these benefits. In recent years, the application of these joints to structural parts constructed from fiber-reinforced composites has increased considerably. Traditional fasteners typically contribute to fiber clipping and, thus, the application of stress concentrations, all of which reduce the integrity of the structure [2].

Two-part adhesives are a fine nominee for binding plastics, polymers, composites, and honeycomb parts together, where interfacial bonding can be compounded by a combination of high vibration, wide temperature differential, and the presence of violent chemicals [3].

**1.2 Bonded Repair**

Compared to the mechanical fastening process, adhesively bonded repair of structures can deliver considerable advantages, including no remarkable weight rise, more consistent stress dispersions, better fatigue behaviour, decreased deterioration, limited shapeshift, simple adherence to specific aerodynamic contours, and reduced care fees. Four standard bonded repairs are usually found: single and double strap and scarf. Because only one side of the structure is often available, and often only one side of a structure is permitted to be fixed for other purposes, in practical applications, one-sided repairs are often implemented. The process includes scraping the affected material and bonding one (single-lap repair) or two patches for the first two cases (double-lap repair). The exterior patch approach is easy to apply, but tensile stretching adds to bending loads due to its eccentricity. Bonding issues are also critical and restrict the applicability of these repairs to non-primary structures. Double-strap repairs mitigate this effect but need access to both of the structure's surfaces. The benefits of quick implementation and low costs are provided by single- and double-strap fixes. However they are not advisable for high-responsibility or heavily stressed systems, so a full-strength recuperation is typically not attained. The patch is inserted in the parent laminate in the scarf and stepped fixes, which allows larger bond lengths and fewer tension condensations owing to the lack of eccentricity of the load. Scarf replacements are distinguished by drilling a conic hole and then adhesively bonding a rising diameter patch layer to fill the area that has been damaged by removing the damaged area. They provide better repair performance compared to lap fixes and have little aerodynamic disruption. The former is stimulated by the removal of the critical joint eccentricities found in strap joints, which act along the loading paths as stress raisers. Due to complications native in the repair process, they present the inconvenience of requiring higher costs, and a large repair area created by the low scarf angles used when high strength values of the repaired joint are needed. In some situations where space constraints exist, this factor can be a disadvantage; also, the growth in adhesive use cannot be ignored in large-scale applications [4].

## 1.3 Epoxy Adhesive

Epoxies are created by polymerizing a combination of two starting materials, the resin, and the hardener. Curing is started when the resin is combined with a determinate catalyst. Curing is the mechanism by which molecular chains, resulting in an exothermic reaction, react at chemically active sites. Covalent bonds between the groups of epoxy resin and the hardener (catalyst) amine groups that derive from this mixture allow for the polymer's cross-linkage and thus determine the epoxy's rigidity and strength.

Temperature monitoring of curing circumstances and choice of resin and hardener compounds helps the mechanical strength properties and thermal, electrical, and chemical resistance to be changed. Therefore, epoxy adhesives have been designed to accommodate a wide variety of diverse applications and working conditions.

Epoxy adhesives conform to a large range of materials, and their features depend on the system's particular chemistry and the nature of the available cross-linking. Remarkable chemical and heat resistance, outstanding adhesion, and water-resistance as well as adequate mechanical and electrical insulation features are some of the most critical performance criteria.

Epoxy adhesives are thoroughly offered as the most commonly used structural form adhesive, either as one part or two-component systems. Epoxy adhesives with one part are normally treatment at temperatures between 250-300° F, terms that engineer a high-strength substance, excellent metal adhesion, and superb environmental and tough chemical toleration. As an alternative to welding and rivets, this tool is also used [5].

### 1.4 Nano Composite Material

Nanocomposites are those composites of which, including nanoparticles, nanotubes, or lamellar nanostructure, one phase has nanoscale morphology.Nanocomposites are those composites of which, including nanoparticles, nanotubes, or lamellar nanostructure, one phase has nanoscale morphology. They have multiphase materials, so multiphase materials should have dimensions in the region of 10-100 nm at least for the phases. Nanocomposites have been designed to offer practical alternatives in order to address the drawbacks of various manufacturing materials today. Based on their distributed matrix and dispersed phase components, nanocomposites can be categorized [6].

Type of material for nanocomposites (as shown in Figure 1.1):

- Nanocomposites of ceramic-matrix
- Nanocomposites of metal-matrix
- Nanocomposites of polymer-matrix

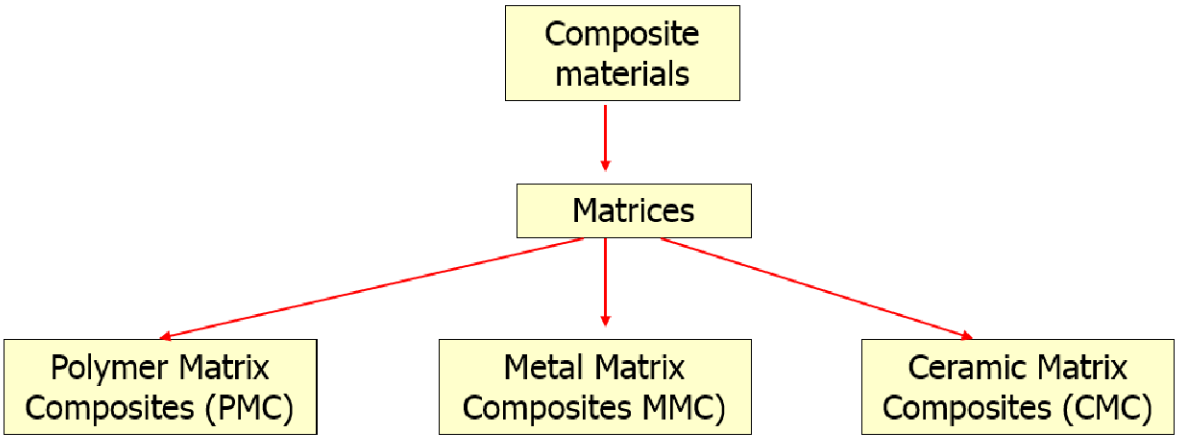


**Figure 1.1** Type of material for nanocomposites.

#### 1.4.1 Ceramic-matrix Nanocomposites

Ceramic matrix composites (CMCs) are a subgroup of composite material. CMCs are made up of fiber-reinforced ceramic fibers immersed in a ceramic matrix-forming ceramic. Currently, CMC's are primarily used in fields that require high-temperature reliability (beyond metal capacity) and corrosion resistance. This contains the space vehicle heat shield device for the manufacture of high-temperature gas turbine components and components for burners and flame holders. Ceramic composites and other composites have several fundamental variations. Ceramic composites and other composites have several fundamental variations. The fibers carry a larger proportion of the applied load of non-ceramic matrix composites. The ratio of fiber and matrix elastic moduli depends on this load partitioning. This proportion can be very high in non-ceramic matrix composites, while it can be as low as unity in CMCs. Another distinctive aspect about CMCs is that the thermal mismatch between components has a very significant effect on CMC efficiency because of reduced matrix ductility and typically high manufacturing temperature [7].

### 1.4.2 Metal-matrix Nanocomposites

Composites of metal matrix (MMCs), as the term suggests, have a metal matrix. Aluminum, magnesium, and titanium are few examples of matrices of such composites. Carbon and silicon carbide are among the common fibers. In order to satisfy the needs of the design, metals are primarily improved to improve or decrease their characteristics. For example, by incorporating fibers such as silicon carbide, the elastic density and strength of metals can be improved, and the high coefficients of thermal dilation and thermal and electrical conductivity of metals can be decreased [8].

### 1.4.3 Polymer -matrix Nanocomposites

Polymer matrix composites (PMCs) composed of a polymer (e.g. epoxy, polyester, urethane) strengthened by fibers of thin diameter are the most widespread developed composites (e.g., graphite, aramids, boron). For example, on a weight basis, graphite/ epoxy composites are roughly five times stronger than steel. The reasons why they are the most widespread composites contain their low cost, high strength, and basic principles of production [8].

## 1.5 Objectives of the Study

- In this work, three different nanomaterials (nano-silica, nano clay, and nanographene) with different ratios will be added to the epoxy resin and their effects on tensile properties of single strap repair of composites laminates will be investigated.

- Effects of patch ratios (D/d=2 and D/d=3) on the tensile properties of single strap repair of composite laminates will be investigated.
- Improvement of the epoxy adhesive with nanomaterials for single strap repairs of composite laminates will be investigated.

## 1.6 Outline of the Thesis

The main stages of this study are divided into five chapters as explained below:

1. Chapter one includes a general introduction about adhesive bonding, bonded repair, epoxy adhesives, and nanocomposite materials, the objectives of the study, and an outline of the thesis.
2. In Chapter two for the mechanical features of composite materials, adhesive bonding of composite structures and single strap repair, a literature review was carried out. Adhesive bonding with nano-particles: nano-clay particles, nano-silica particles, nano graphene particles.
3. In Chapter three, information about the materials and tools production of test examples, and experimental tests.
4. In Chapter four. presents the results of tensile tests to show the effect of nanomaterials nano-silica, nano-clay, and nano-graphene with epoxy resin on the single strap repair and discussed.
5. Chapter five includes a conclusion on the experimental analysis.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 Introduction

In this chapter, a literature review has been conducted for the mechanical features of composite materials on the adhesively bonded repair techniques. Many authors have studied the mechanical features of composite materials on the adhesively-bonded repair techniques. This study examines the mechanical properties of nanocomposite materials in epoxy.

## 2.2 Adhesive Bonding

The materially joined systems are delegated to adhesive-bonding. Bonding methods serve to create material joints of the same type of mixture of materials. There are many advantages and disadvantages of adhesive bonding repairs.

### 2.2.1 Advantages of Adhesive Bonding

Comparison of some processes of joining is given in Figure 2.1.

- The adherents are not debilitated by bores as it is the case, for example, when screwing and riveting. Power transfer is therefore surface-related rather than spot-related (Figure 2.2).
- High temperatures, as in welding and, in part, even in soldering, do not stress adherents there. Thermally mediated alterations to material properties are thus discouraged, allowing heat-sensitive materials to be joined together.
- Adhesive bonding makes it possible to bond very diverse materials with themselves or other materials while maintaining their individual characteristics. In the above case, the various beneficial properties of revolutionary composite systems can be used.

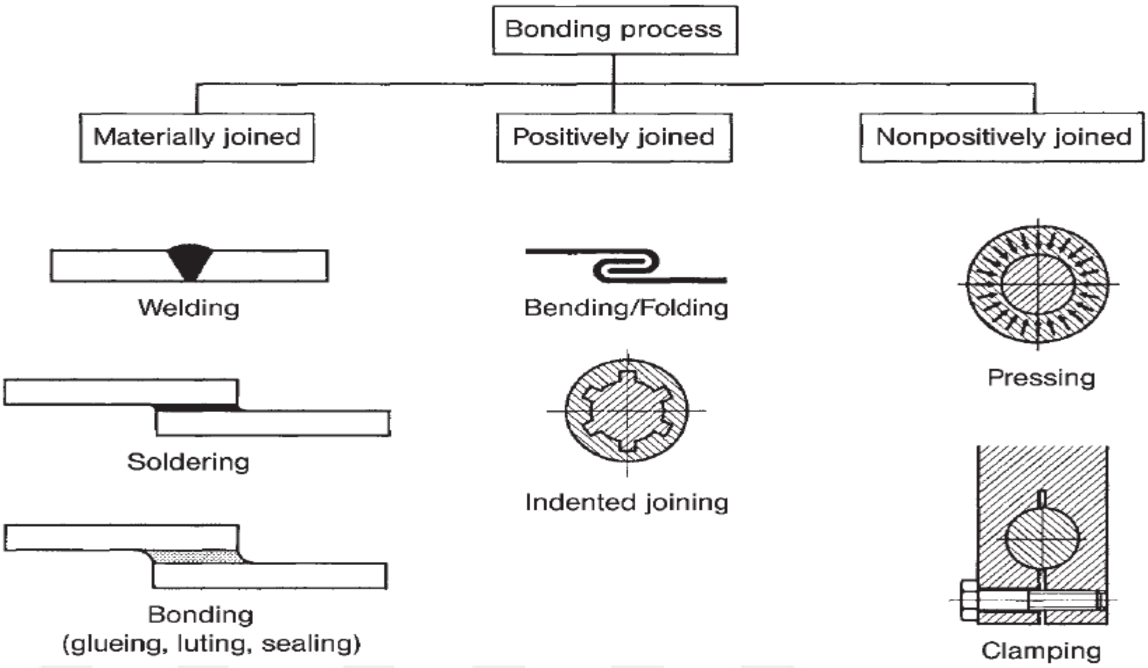


**Figure 2.1** Classifying the mechanisms of joining [9].

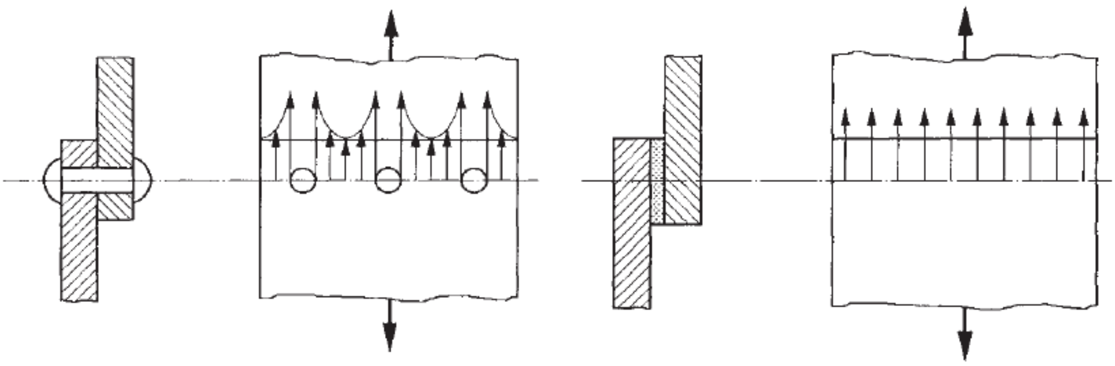

**Figure 2.2** Transmission of power into riveted (screwed) and bonded joints [9].

- Bonding as a joining mechanism allows very thin materials to be joined (< 500 μm). This technique is especially important for lightweight building manufacturing and the resulting weight reduction (including aerospace manufacturing). In addition, it is the basis of an increasingly varied packaging industry architecture of film-type laminates.
- Homogeneous stress dispersion when stress-loading, relative to riveted or screwed links (Figure 2.2).

### 2.2.2 Disadvantages of Adhesive Bonding

- The adhesive layer's heat resistance is limited. Based on the general definition the adhesive content, temperatures for the continuous stress range between approximately 120 and 300 °C.
- Environmental effects, such as humidity, may affect adhesive layers and their boundary layers against the adherent surfaces, resulting in a decrease of strength.
- The development of bonded joints includes surface preparation of the adherents as an extra processing stage, with a couple exceptions (e.g., body-in-white production).
- In the manufacture of bonded joints, the time needed for the relevant curing of reaction kinetics must be taken into account.
- The increasing need for recyclability of industrial materials demands adequate design-engineering steps to be taken.
- There is a very restricted availability of non-destructive test methods [9].

For centuries, adhesives and sealants have been used, with natural materials such as tree sap, beeswax, and tar making up the earliest adhesives and sealants. Subsequently, refined natural products such as animal protein, resin and natural latex were used. The array of adhesive and sealant formulations has exploded with the advent of the chemical industry and synthetic polymers. The adhesives and sealants of today can be categorized in several distinct ways, such as by method of cure (bonding), form of chemistry, and even implementation (e.g. structural vs. non-structural) [10]. Thanks to their better wetting capability, outstanding mechanical properties, and good chemical and thermal resistance, epoxies dominate the field of structural adhesives. However, high fragility and high-water absorption capacity are present in fully curated epoxy resins, reducing their use in humid conditions [11].

Commonly used adhesive bond weakened structure strategies consist of combinations of single or double-lap/strap, scarf, and step (Figure 2.3 shows the schematic depiction of single-strap, double-strap, and scarf repairs). The benefits of quick application and low costs are provided by single and double-strap fixes. For high-responsibility systems, though, they may not be feasible because these geometries contribute to moderate concentrations of peel and shear stress at the bond edges, rising from the various straining effects on the structure and patches that hinder the high efficiency of these repairs [12-13].

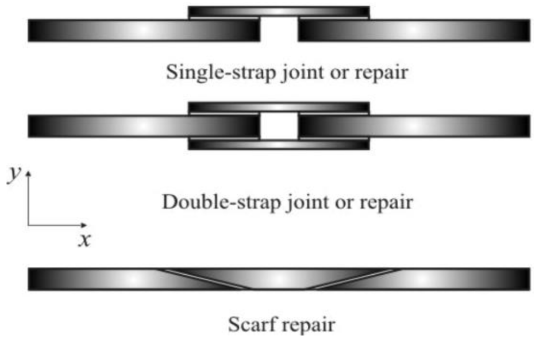


**Figure 2.3** Schematic view of single-strap, double-strap, and scarf repairs.

De Moura [14] was investigated the implementation of unified region modelling to composite bonded repairs. It was concluded that a combination of patch internal chamfering and 45° flat fillet consists of the strongest combination of the geometrical results studied. Daraker and Chandore [15] have researched the effect of surface shagginess on single lap adhesive joint strength. During the design stage of adhesively bonded joints, the surface roughness parameter was found to be considered, as the bond strength differed greatly by 30-35 percent between the various surface roughness values. Cheng et al. [16] have investigated the study of an adhesively bonded single-strap joint combined with shape memory alloy (SMA) reinforced layers. It was concluded from the numerical analysis reveals that the SMA ensures an efficient means for the load carrying capability of composite joints to be increased. Tsai et al. [17] have researched the stresses in double-lap adhesive joints with laminated composite adherents. Experimental and finite elements demonstrate that owing to the three-dimensional phenomenon, the displacement fields are obtained from both analyses, respectively, for both joints, with the exception of fringe waviness. Çitil [18] investigated the effects of patch material on the adhesive in the repair of broken pipes. It was concluded that it is considered that the repairing of damaged pipes exposed to internal pressure, the patch of the type of material, and the angle of the overlap are of great importance. Whittingham et al. [19] have researched the micrographic experiments on adhesively bonded scarf repairs to the thick composite air structures. Two strategies were researched for generating the hard-patch. The first was the molded technique, where before bonding in the repair cavity, the patch was laid up in a mold and cured. It explains the development and application of the molded hard-patch repair technique on a horizontal F/A-18 stabilizer. The second solution includes machining the patch from a composite panel to get the scarf emptiness surface using digitized data collected from the use of surface profiling equipment. In order to evaluate essential features of the bond-line created from the various techniques, micrographic techniques were used. It was concluded that the test both the easy and hard-patches exhibited several features which considerably varied from the ideal scarf configuration. Any of these traits, such as ply aberration in the patch body, are a problem, others may not be a concern, such as lack of ply convergence along the scarf in the case of the soft-patch and folding the ends of the ply throughout the surface of the scarf cavity. Pinto et al. [20] have studied strap repair optimization by using embedded patches. In this work in order to allow geometry optimization by adjusting the overlap length, regular single strap and double strap repairs and also with embedded patches in the adherents were checked under strain, thereby allowing the strength of the repair to be maximized. It was concluded the yielding properties and measurements of the patches and adherents (e.g. thickness) have a direct effect on the strength of the repair, and the plastic deformation of the adherents and patches greatly decreases the repair performance for large overlaps. If, for a given value of overlap time, plastic deformation of the patches and/or adherents occurs, further increasing this parameter does not yield a strength recuperation of repairs. Okafor et al. [21] investigated the design analysis, and enduringness of adhesively bonded composite patch repairs of chapped aircraft aluminum panels. As a research specimen, pre-cracked 2024-T3 coated aluminum 381 · 89 · 1.6 mm (15 · 3.5 · 0.063 in.) panels mended with an octagonal single-sided boron/epoxy composite patch were used. Two separate composite ply configurations have been investigated, 5-ply and 6-ply. On the test specimen, linear and non-linear finite element tests were accomplish using 8-noded hexagonal elements of 24 degrees of freedom (DOF). The stress distributions obtained were used to estimate the increase in the restored structure's intensity and longevity. There was a comparison of the stress values at critical stages. It was concluded that after the application of the patch, maximum skin stress reduces dramatically. For the patched specimen, the maximum skin stress is decreased by 83-85% relative to that of the unpatched specimen. Related findings are seen from linear analyses of both 5-ply and 6-ply, while non-linear analyses indicate that 6-ply patch mend has lower peak skin stresses than 5-ply patch mend. Meier [22] has studied demonstrate how advanced polymer matrix composite materials evolved for high-performance aircraft can deliver significant advantages for repairing substructures. It was found that “non-laminated CFRP straps’’ will play a key role in bridge post-strengthening, offering universal, easy to anchor, and inexpensive bridge repair tensile components. Manalo et al. [23] investigated mechanical tests of tensile, double strap shear connection prototypes and structural testing of rehabilitated I-beams will assess the pre impregnated carbon fibre reinforced epoxy repair system. It was found that due to the improved consolidation of the fiber layers, the use of a vacuum during laminate processing led to better mechanical properties. A 120 mm bond length provides a more consistent and durable adhesive bonded joint and is representative of the rehabilitated steel beam's bond strength, the virtual crack and corrosion defects in steel I-beams have been successfully repaired by a patched carbon prepreg device, restoring them to their original load bearing capability and stiffness. Khan and Kumar [24] have studied a stress-function technique to analyze the stress-state in an adhesively bonded single-sided composite patch-repair system featuring a through-thickness hairline defect in the substrate sustaining far-field tensile stresses. It was concluded that using a material tailored bond line, the peak adhesive stresses can be significantly reduced and distributed more uniformly across the bond length, and the proposed model can be used as a simple and reliable analytical tool to design such composite crack-patch systems and to evaluate the impact of interface stiffness failure due to an existing bond line defect and/or damage. Her and Chao [25] investigated that the use of adhesively bound patches to restore damaged composite laminates. To analyze the stress distribution in the bonded region, a particular adhesive component is made. The standard elements in the laminate and patch may be inserted using the adhesive part. It was found that a stiffer and thicker patch can hold higher loads and, as a result, decrease the load around the damaged region, resulting in less stress concentration in the damaged hole, and less loads are moved to the patch with a high shear module and thin adhesive layer thickness, resulting in a high stress concentration in the damaged hole. Wang et al. [26] investigated the analysis of the damage forbearance of adhesively bonded repairs to composite structures. In order to examine the impact of pre-existing defects on the load carrying capability of a scarf joint, a number of tests have been undertaken using specimens of various disbond lengths. The load carrying ability of the scarf joint was observed to decrease with the size of the initial defect, at a rate faster than the decrease in the bond area. The cohesive model using adhesive properties has been found to over-predict the joint strength, underestimating the strength reduction due to initial flaws. The load-carrying capacity of the scarf joint has been found to decrease with the size of the initial flaw, at a rate faster than the reduction in a bond are cohesive model using adhesive properties has been found to over-predict the joint strength, underestimating the strength reducing due to initial flaws. Chester et al. [27] have studied adhesively bonded repairs to the primary aircraft structures. In order to produce design data for the fatigue durability of the adhesives used in bonded repairs, a test sample was developed, and both shear strain range and strain energy release rate were investigated as parameters to characterize the behavior of fatigue. This detail was used in the creation of a 48 mm long crack adhesively bonded composite repair in the lower wing skin of a F-111 aircraft. It was found a fatigue crack has been successfully fixed with an adhesively bound boron/epoxy patch in the lower wing surface of a RAAF F-111 aircraft. The critical nature of this crack has necessitated an extensive validation program for this repair. A detailed FE analysis in conjunction with three levels of structural testing has validated the repair by independent means. Shear strain in the adhesive bond line appears to be a more promising parameter than strain energy release rate for fatigue design. Charalambides et al. [28] investigated the output under static and fatigue loading of carbon fiber/epoxy patch joints, bonded with an epoxy film adhesive. For periods of up to 16 months, the repair joints were submerged in distilled water at 508C and the influence of the hot/wet atmosphere was assessed on the static and fatigue strengths. Residual strength checks were also conducted, where repairs were subject to fatigue accompanied by static loading. It determined the mechanical features of the substrate and the adhesive forming the joint. At room temperature, both experiments were performed. It was found that there was no noticeable influence of the conditioning on the above properties and that the repair joints had a similar static strength to that of the parent material. Caminero et al. [29] have investigated monitoring of damage and analysis of open hole composite laminates and adhesively bonded repairs using digital image correlation. In the present work, the calculation of the damage resulting in composite plates with an open hole when loaded in tension using Digital Image Correlation (DIC), a full-field optical strain measurement, has been investigated. In addition, the DIC device was used to evaluate damage and the efficiency of adhesively bonded patch repair in composite panels under tensile loading. It has been used effectively to quantify surface strains and possible damage to the composite structure that may occur. The strain and displacement measurements were compared in good agreement with the analytical estimates. The latest work has shown DIC's ability for on-line monitoring of composite structures. Banea and Silva [30] have researched fiber reinforced plastic (FRP) composite structure adhesive bonded joints (single skin and sandwich construction). For adhesively bonded FRP composite structures, the impacts of surface preparation, joint configuration, adhesive properties, and environmental factors on the joint behavior are briefly defined. Adhesive bonding has been found to be a viable technique for combining composite materials, although the joint performance is limited by low interlaminar shear and tensile strength. Suitable surface treatments and adhesives have to be selected for a specific application. Normally, the preference of the adhesive is best determining the type of composite to be bonded, the application, the operating environment, and the expense. Baker and Chester [31] investigated minimum surface treatments on repairs that are adhesively bonded. It has been observed that the environmental durability of adhesive bonds in both epoxy and acrylic adhesives is improved by the use of a silane solution together with a primer. Silane plus primer treatments can be used to achieve longevity similar to that of acid anodization treatments, along with significant savings in surface treatment speed an essential consideration during aircraft repair. The compatibility of various adhesives and primers should be experimentally determined, as this study has found differences in the efficacy of primers in improving bond strength and durability. l'Armée et al. [32] have investigated the study deals with the theoretical determination of section forces and single lap moment joints with composite laminate adherents, including extensional coupling bending. the new method for determining the bending moment and shear forces was found to be proposed in single lap joints with composite adherents, which allows the study of joints with a symmetrically laminated adherend displaying extensional bending coupling. The model refers to different types of boundary conditions (simply supported ends, fixed ends, repair brace, bonded doubler) and it is possible to determine a solution for the bending moment with very low computational effort. Pandey and Kumar [33] have researched numerical studies of the interface behavior of the substratum of adhesively bound cracked aluminum alloy patched with composite material lined with fiber. The peel and shear stresses were found to be prevalent and the peak shear stress resides in the adhesive at the edge of the patch. In this analysis, different stress and strain-based failure parameters were considered to the strength. Prolongo et al. [34] have investigated a comparative research on the adhesive characteristics of various epoxy resins. They also observed that coherent fracture mechanisms have major involvement. The expansion of these micro-cohesive structures has also been found to be closely associated with the strength of the adhesive. The two distinct processes of crosslinking, addition reaction, and homo-polymerization are based on studied epoxy formulations.

### 2.3 Single Strap Repair

Khan and Kumar [35] have investigated interfacial stresses with a material optimized bond line in single-side composite patch-repairs. They observed that the peak adhesive stresses can be greatly reduced and that using a tailored material bond line instead of a homogeneous bond line, they can be distributed more uniformly over the length of the bond. Bulut et al. [36] studied with the use of micro-scale perlite and sewage sludge ash (SSA) fragments for glass-epoxy laminates, the tensile properties of epoxy adhesive include adhesively bound single-strap repairs. The joining efficiency of composite laminates was found to be enhanced by combining perlite or SSA filler with epoxy adhesive at a low percentage of the weight of both fillers, leading to solving major engineering problems during operation. Campilho et al. [37] have studied an experimental and finite element parametric for the behavior of single and double-strap repairs of carbon-epoxy laminates under buckling unrestrained compression. It was concluded that the finite element approach can be a powerful statistical method and a choice for the diminution of fees due to experimentation, assuming that sufficient parameters are used to model the various forms of fractures. Moreira et al. [38] have studied high-cycle fatigue analysis of single-strap repairs of carbon– epoxy composite laminates. It was concluded that the cohesive failure within the adhesive caught out greater fatigue life compared to the interlaminar failure of the adherent and applied force is the most significant parameter influencing the fatigue life. Wang et al. [39] have investigated the research on sided bonded repair of out-of-plane bending in. In an unsupported one-sided repair, they observed that localized bending of the reinforcement can cause a substantial increase in the stress strength factor, thereby decreasing the repair performance, and parametric experiments have shown that the most efficient way to minimize the out-of-plane bending effect is to use thicker reinforcement. Shahin and Taheri [40] have studied the analysis of deformations and stresses in single-strap joints with balanced and unbalanced adhesive bonds. It was the demonstrate that strap joints, as long as they are correctly built, can be as powerful as lap joints. The derived solutions offer deeper insight into understanding the parameters that affect the edge forces most.

## 2.4 Adhesive Bonding with Nano-Particles

Nanomaterials are used to strengthen composite materials. Pattanaik et al. [41] investigated the effect of the mixing time on the mechanical features of epoxy-based composites filled with fly ash showed that adequate combining between adhesive and filling particles is important. Srivastava [42] has investigated the composites with identical carbon/carbon (C/C) and carbon/carbon-silicon carbide (C/C-SiC) substrates were bonded with unmixed epoxy resin and the one containing 3% multiwall carbon nanotubes (MWCNTs). The results of tests show that C/C-C/C and C/C-SiC-C/C-SiC substrates bonded with MWCNT/filled epoxy resin have a greater adhesive joint strength than those bonded with epoxy resin only. Jojibabu et al. [43] have been investigated the effect of various carbon nano-fillers on rheological features and lap shear strength of epoxy adhesive connections. It was found with the inclusion of carbon nano-fillers, a thermo-gravimetric study revealed an improvement in the thermal stability of the epoxy. Increased lap shear resistance with a high Weibull modulus resulted in carbon nano-fillers. Tutunchi et al. [44] have studied the effect of $TiO_2$ nanoparticles on the adhesion strength of composite joints of steel-glass/epoxy bonded with two-part acrylic structural adhesives. The shear and tensile strengths of the adhesive joints have been shown to improve with the addition of up to 3 percent of the filler material, during which the addition of more filler content has reduced shear and tensile strengths of the adhesive connections. A decrease in the peel power of the connections was also caused by the incorporation of nanoparticles.

## 2.5 Nano Clay

Ho et al. [45] have researched the mechanical features of composites based on epoxy using nano clays. It was found that compared to an unmixed epoxy sample by an improvement of around 5%, the formulation at 5 wt. percent nano clay gave a higher ultimate tensile power. In addition, it gave among all the compositions the largest vickers hardness rating. The ductility decreased significantly, however, and right after showing the level of ultimate tensile strength, the sample was suddenly separated.

Khalili et al. [46] have studied nano clay strengthened epoxy adhesive bonded connections made of composite materials have mechanical properties. The content of nano clay was 1, 3, and 5 percent of epoxy resin (Araldite LY5052) 2). The adhesive joints with 1 percent nano clay particles were found to have the greatest tensile load strength and the highest values of charpy impact energy were found for adhesive joints loaded with 3 percent nano clay particles. Kaboorani and Riedl [47] have studied the effects of nano-clay addition on polyvinyl acetate (PVA) performance as a wood adhesive. They also observed that, by applying nano-clay to PVA, the shear strength of wood connections improved in all states. Depending on nano-clay loading and form, the addition of nano-clay has developed the thermal steadiness of PVA to varying degrees. Morphological studies have shown that the fluctuations found in nanocomposite thermal stability and bond strength derive from nano-clay dispersion quality.

## 2.6 Nano Silica

Zhou [48] has studied the adhesive features of nano-silica epoxy-bonded single lap joint. It was found that compared to pure epoxy, the adhesive strength improves by 20 percent under quasi-static loadings. Tutunchi et al. [49] investigated the effect of silica nanoparticles on the adhesion ability of composite joints of steel-glass/epoxy bonded with two-part acrylic structural adhesive. They observed that differential scanning calorimeter analysis showed that the adhesives Tg values improved with the nanofiller content increasing. For adhesives including nanoparticles, the equilibrium water touch angle was diminished. The incorporation of nanoparticles changed the fracture morphology from smooth to rough fracture surfaces, scanning electron microscope micrographs showed. Hassanifard and Paygozar [50] have investigated in five concentrations, the effect of nano-silica nanoparticles on the bonding strength of reinforced adhesive joints has been experimentally tested. They also discovered that a feature of nanoparticle concentration is the change in bonding strength. In inclusion, the addition of silica nanoparticles was concluded to have a suitable impact on the joint strength at an optimal point in which the joint strength exceeds its maximal value, and a further rise in the weight fraction of the nanoparticles allows the joint strength to decline.

He et al. [51] have studied the effects of the nano-silica material on the structures of Ti-epoxy assemblies and epoxy nano composites. It was found that although with the rise in silica addition, the glass transition temperature of nanocomposites decreased, the mechanical properties of nanocomposites such as flexural strength, fracture resilience, and stiffness improved to the maximum values when the silica content was 15 percent and the silica content increased, the shear strength steadily decreased. With 2.5 wt. percent silica inclusion, the extraordinary improvement in shear strength can be due to the increased wettability between the epoxy and Ti surface, which resulted in a much higher bonding strength between them. Razavi et al. [52] studied the single lap joints bonded with structural adhesives reinforced with a mixture of silica nanoparticles and multi-walled carbon nanotubes. They observed that there was an important influence on the mechanical activity of SLJs by including the mixed nanoparticles. Among the various weight percentages of the mixed nanoparticles tested, the largest increases in shear strength and elongation at failure were 28 percent and 36 percent, respectively, which were related to 0.8 percent of the mixed nanoparticles. In addition, owing to the addition of mixed MWCNTs and SNPs, the increase in mechanical properties was greater than that gained for corresponding single-type nanoparticles of the same weight percentages.

### 2.7 Nano Graphene

Khoramishad et al. [53] have investigated the impact of nano-platelet graphene oxide (GONPs) on the performance of nanocomposite adhesive joint measured at elevated temperatures. It was found that the average thickness of the flakes of sGO was larger than that of the flakes of GO. 10 mm diameter patch holes were opened by a CNC machine. After the inclusion of a sGO that included amine groups, the bonding strength of a carbon fiber/epoxy hybrid, measured with a single lap joint bonded with an epoxy adhesive, was improved by 53 percent. These findings show that sGOs can improve the interfacial bonding between the carbon fibers and the epoxy adhesive, especially those containing amine functional groups. Guadagno et al. [54] investigated the production of a new formulation of graphene nano-modified to increase the mechanical strength of structural adhesives. At a concentration of up to 1% wt., they find the epoxy adhesives loaded with graphene greatly improved the mechanical behavior of the bonded joints. The addition of 4 % wt. graphene has no major impact on mechanical efficiency only in the case of non-filled adherents. This is likely attributable to nanofiller agglomerations that induce heterogeneity at the interface between adherents and adhesives in large domains. The effect of the integration of graphene into the adherents, which has an effect on the chemical stability between adhesive and adherent surfaces, has contributed to a significant improvement in tensile strength relative to the corresponding unfilled adherent joints. Due to the combined effects of intermolecular interactions between graphene platelets and the resin network, this beneficial effect is very possible. Daliri et al. [55] have investigated using graphene nanoplatelets, structural health testing of faulty single lap adhesive joints. The findings revealed that the functionalized adhesive of graphene nanoplatelets (f-GNP) is susceptible to deformation and extension of the adhesive layer injury. By integrating the 12 percent f-GNP into the adhesive joint without an incorporated defect, the ultimate intensity has risen by about 74 percent. Pawlik et al. [56] have investigated the effects of surface alteration and graphene nanoplatelet reinforcement on the aluminum alloy adhesive joint. It was found with the insertion of 0.3 percent graphene oxide nano-platelets (GOPs) into the adhesive sheet, the maximum improvement of 69 percent in the fracture energy was obtained. Moreover, to figure out the toughening processes involved in this enhancement, the fracture surfaces were analyzed using the scanning electron microscopy method, and mechanisms of GOP debonding and crack deflection were found.

## 2.8 Conclusion

- According to the literature review adhesive bonding, technic is open to improvement.
- Adhesive-bonding makes it possible to join incredibly distinct materials with themselves or other materials while maintaining their unique characteristics.
- Adhesive layers and their boundary layers to the adhesive surfaces can be affected by environmental effects, such as moisture, resulting in a decline in strength.
- Many different materials have been used for the bonded repair, these are nano-silica, nano-clay, nano-graphane, fly ash, ZnO, and TiO.
- Nanomaterials have been used to enhance mechanical features and environmental resistance of adhesives.

- Single and double-strap repairs put forward the advantages of simple application and low fees. For high-responsibility systems, though, they may not be feasible because these geometries contribute to moderate concentrations of peel and shear stress at the bond edges, increasing from the various straining effects on the structure and patches that obstruct the high efficiency of these repairs.
- As seen in the literature review, there is no study of the mechanical properties of epoxy bonding with nanomaterials on single-strap repair of glass epoxy laminates, so it is a subject open to improvement.

# CHAPTER 3

# EXPERIMENTAL STUDIES

## 3.1 Introduction

This section explains the properties of the materials used in the single strap repair experiment and their preparation.

## 3.2 Materials

### 3.2.1 Tools

The tools were used in the experiment are as follows:

- Universal tensile machine: used to perform mechanical tests on samples
- Geotine machine was used for cutting S-Glass fiber reinforced (SGFR) composite materials.
- CNC machine was used for drilled S-Glass fiber reinforced composite materials.
- A digital scale was used to adjust the epoxy, hardener, and nanomaterials ratio.
- The mixer was used for stirred the epoxy, hardener, and nanomaterials ratio.
- Sandpaper was used to smooth the surface of the composite plates and patches.
- Acetone was used to clean the composite plates and patches.

The tools are shown in Figure 3.1.

a)


b)

c)

d)

e)

f)


g)


**Figure 3.1** The tools in the experiment.

### 3.2.2 Composite Plates

Due to some desirable features such as low density, high special strength, and rigidity, S-Glass fiber reinforced (SGFR) composite materials have been used thoroughly in the aerospace, civil and structural industries. The composite plates have used in the experiments were the density of SGFR is 200 g/$m^2$ and 2 mm thickness. SGFR plates commercially produced were bought from the Kupar Pompa, Küçükparmak Mühendislik San. Tic. Ltd. Şti. The main mechanical properties of S-glass fiber are listed in Table 3.1. Composite material used is shown in Figure 3.2.

**Figure 3.2** The composite plate.

**Table 3.1** Mechanical properties of S-glass fiber [57].

| **Mechanical data of glass fiber (plates and patch)** | | | |
|---|---|---|---|
| **Property** | Test | Unit of Measure | Value |
| **Tensile Strength** | ASTM D638 | psi | 45,000 |
| **Flexural Strength** | ASTM D790 | psi | 60,000 |
| **Compressive Strength** | ASTM D695 | psi | 65,000 |
| **Hardness** | ASTM D785 | NA | Rockwell M110 |
| **IZOD Impact-Notched** | ASTM D256 | ft-lb/in | 14 |

### 3.2.3 Nano-Silica Particle

Silica is the generic term for silicon dioxide (SiO2) compound materials which appears in crystalline and amorphous shapes. In different ways, crystalline silica occurs. Quartz is a popular and well-known material, and more simply, a-quartz. A-quartz is converted to b-quartz, tridimite, and cristobalite when heated. The family name of porous crystalline silica is porosil. In natural and synthetic forms, quartz occurs, while all porosiles are synthetic. It is possible to separate amorphous silica into natural samples (e.g. diatomaceous earth, opal, and silica glass) and items produced by humans [58].

In a number of industries, the implementation of synthetic amorphous silica, particularly silica nanoparticles (SNPs), has admitted great attention. SNPs are processed as additives to cosmetics, medications, printer toners, varnishes, and food on an industrial scale. For a host of biomedical and biotechnological implementations, such as cancer therapy, DNA transfection, drug delivery, and enzyme immobilization, nano-silica is also being developed [58].

It is possible to condense silica from vapors produced during volcanic eruptions. In living species, natural silica may also be deposited from supersaturated natural water or polymerized (biogenic silica). Such amorphous biogenic silica can be present in multiple living organisms as discrete fragments, skeletal components, or surface elements. Many microcrystalline silica crystals, such as flint, chert and chalcedony, are obtained by compaction from biogenic silica after crystallization [58]. Nano silica particles are shown in Figure 3.3, and chemical compositions of the silica are viewed in Table 3.2.

**Figure 3.3** Nano silica particles.

**Table 3.2** Chemical compositions of the silica [59].

| **Chemical Composition** | |
|---|---|
| **Content** | **Nano Silica** |
| $SiO_2$ | 99.05 |
| $Al_2O_3$ | 0.05 |
| $Na_2O$ | 0.48 |
| LOI | 0.1 |
| **Physical Properties** | |
| Specific Gravity | 1.37 |
| Specific surface area(BET) $m^2/g$ | 85 |

### 3.2.4 Nano-Clay Particle

Nano-clay is a product made up of phyllosilicates, which are compose dependent on oxygen, silicon, and other elements and which are chemically pretreated and degenerated from natural sources. In various products, Nano-clay is used after further processing steps. They will swell or shrink when water accumulates or is removed between layers due to their layered nature. Nano-clay volumes can be increased up to six times by water absorption and stable gels can be formed. As a result, nano-clay has special properties that can be used in multiple industries, such as turning desert sand into fertile soil [60].

Nano-clay comprises of phyllosilicates containing mineral groups such as talc, mica, kaolin, montmorillonite, serpentine, and sepiolite. Nano-clays vary in the size and sequence of the areas in which the SiO4 tetrahedra in the layers are directed upward or downward, among other aspects. Additionally, nano-clay varies from the incorporated ions. It has been demonstrated that only mild and temporary inflammation in the lungs is caused by inhaling nano-clay particles. There is little information to date about how nano-clay performs in the field [60].

As the main constituent of bentonite, montmorillonite, the most scientifically important clay mineral, consists of SiO4 tetrahedron bilayers of deep-rooted octahedral layers of aluminum, iron and hydroxide ions. A typical montmorillonite particle comprises aluminosilicate layers of approximately 1 nm thickness with lateral sizes in the 700 nm to approximately 10 μm series that accumulate into large stacks [60].

Figure 3.4 shows nano clay particles, and table 3.3 shows the chemical composition of the nano clay particles.

**Figure 3.4** Nano clay particles.

**Table 3.3** Chemical composition of the clay [61].

| Chemical Compound (Oxide) | Montmorillonite |
|---|---|
| $Na_2O$ | 1.13% |
| CaO | 1.02% |
| $Al_2O_3$ | 18.57% |
| $SiO_2$ | 43.77% |
| $H_2O$ | 36.09% |

### 3.2.5 Nano-Graphene Particle

The one-atom sheet of pure carbon is graphene. It is one of the carbon changes termed (crystallographic) alterations. Despite the same chemical formula, two changes exhibit distinct properties since the carbon atoms are arranged differently (see crystal structure). The graphene layer thickness is approximately 0.3 nanometers and this amount is approximately one hundred thousandth of the thickness of the hair of the human scalp. Very commonly, the lateral distribution of a sheet is much larger. Each carbon atom is chemically bound to three other carbon atoms in a graphene monolayer. This leads to a structure of honeycomb layering that is (two-dimensional) [62]. Graphene is an almost translucent material that has the electrical conductivity of any known substance at the greatest room temperature. The atoms of Graphene are arranged in a hexagonal structure. Graphene is also one of the hardest materials in the known universe, although it is a mere one-atom thick, the only two-dimensional substance. Graphene is more than 200 times stronger than steel, with a tensile strength of 130 GPa. On top of that, graphene is an exceptional heat and electricity conductor and has interesting abilities for light absorption. Scientists and engineers thus expect that several potential devices, such as ultra-high-speed transistors and transparent electrodes, touch screens (for LCD or OLED displays), solar cells, etc., will be realized using graphene [63]. Figure 3.5 shows nano-graphene particles, and Table 3.4 shows the chemical composition of the nano graphene particles.

**Figure 3.5** Nano-graphene particles.

**Table 3.4** Chemical composition of the graphene [64].

| Chemical Compound (Oxide) | Percent weight (% wt.) | Standard Deviation (σ) |
|---|---|---|
| Carbon (C) | 48,35 % | ± 6,12 |
| Oxygen (O) | 23,60 % | ± 6,40 |
| Others (H,S,N) | 28,05 % | ±12,50 |
| C/O | 2,11 % | ± 0,34 |

### 3.2.6 Epoxy Resin and Hardener

An epoxy resin is a type of thermoset polymer formed from a monomer containing at least two classes of epoxides. It is possible to homopolymerize or cross-link epoxy resins into a tri-dimensional network. For atmospheric or thermal-curing, a wide variety of curatives are available that involve polyfunctional acids, amines, phenols, thiols, alcohols, and anhydrides, and are sometimes referred to as curing agents or hardeners [65].

For a variety of uses, epoxies may be used. One of the most common uses is arguably using epoxy for adhesive applications. They are considered the best usable adhesive, and in the automobile and aerospace industry, epoxy adhesives have seen application. Water resistance, toughness, chemical resistance, and thermal resistance are given by solvent-free epoxy adhesives [65].

Where heavy duty service is required, epoxy coatings find use on metal substrates. They have a hard protective coating of superior toughness [65].

In this experiment, an epoxy resin (MOMENTIVE-MGS L285) with a hardener (MOMENTIVE-MGS H285) at a stoichiometric ratio of 100:40 was used as the matrix as shown in figure 3.6. The main mechanical properties of epoxy resin and hardener are listed in Tables 3.5 and 3.6.

a) b)


**Figure 3.6** Hardener and epoxy.

**Table 3.5** Mechanical features of epoxy resin [66].

| | |
|---|---|
| **Density** | **1.18 -1.20 [g/cm³]** |
| **Flexural strength** | **110-120 [N/mm²]** |
| **Modulus of elasticity** | **3.0-3.3 [N/mm²]** |
| **Tensile strength** | **70-80 [N/mm²]** |

**Table 3.6** The hardener's mechanical features [67].

| | |
|---|---|
| **Density (gr/m$^3$)** | **0.94-0.97** |
| **Viscosity(mPas)** | **50-100** |
| **Amine Value (mgr KOH/gr)** | **480-550** |
| **Refractor index** | **1.5020-1.500** |
| **Measuring Temperature** | **25 C°** |

### 3.2.7 Sandpaper

Sandpaper is manufactured in a number of grit sizes and is used either to strip material from surfaces and make them cleaner (in painting and wood finishing, for example), to eject of coating of material (such as old paint), or often to construct the surface uneven (for example, as preparation for gluing). When identifying the document, it is popular to use the name of the abrasive, e.g. "aluminum oxide paper", or "silicon carbide paper" [68].

The sandpaper grit size is commonly specified as a number that is reversely proportional to the size of the particles. A little number of 20 or 40 exposes a coarse grit, while a big number of 1500 shows a fine grit [68]. The sandpaper used is shown in Figure 3.1.

### 3.2.8 Acetone

A solvent applied in the manufacturing of plastics and other consumer products is acetone, a colorless substance commonly known as propanone. Acetone can also be applied to a small degree in household items, containing cosmetics and personal care products, where nail polish remover formulations are most commonly used. As a byproduct of metabolism, acetone happens naturally in the human body [69]. Acetone used is shown in Figure 3.1.

Acetone is also formulated as a solvent into solvent systems or 'blends' used in the formulation of lacquers for automobile and furniture finishes. To decrease the viscosity of lacquer solutions, acetone can also be used. Acetone is widely used in the textile sector for degreasing wool and degumming silk [69].

### 3.3 Production of Test Examples

Firstly, the composite plates were cut to using a Geotine machine. The size of the composites plates was 100 x 50 mm. The middle of the composite plates was perforated in a CNC machine with a diameter of 10 mm, resulting in a patch repair ratio of D/d=2 and D/d=3 (patch diameter (D) and hole diameter (d)). For the patch ratios, the patch diameters used were 20 and 30 mm. The overall sample thickness was 2 ± 0.2 mm. The composite plate which is cut and drilled is shown in Figure 3.7.

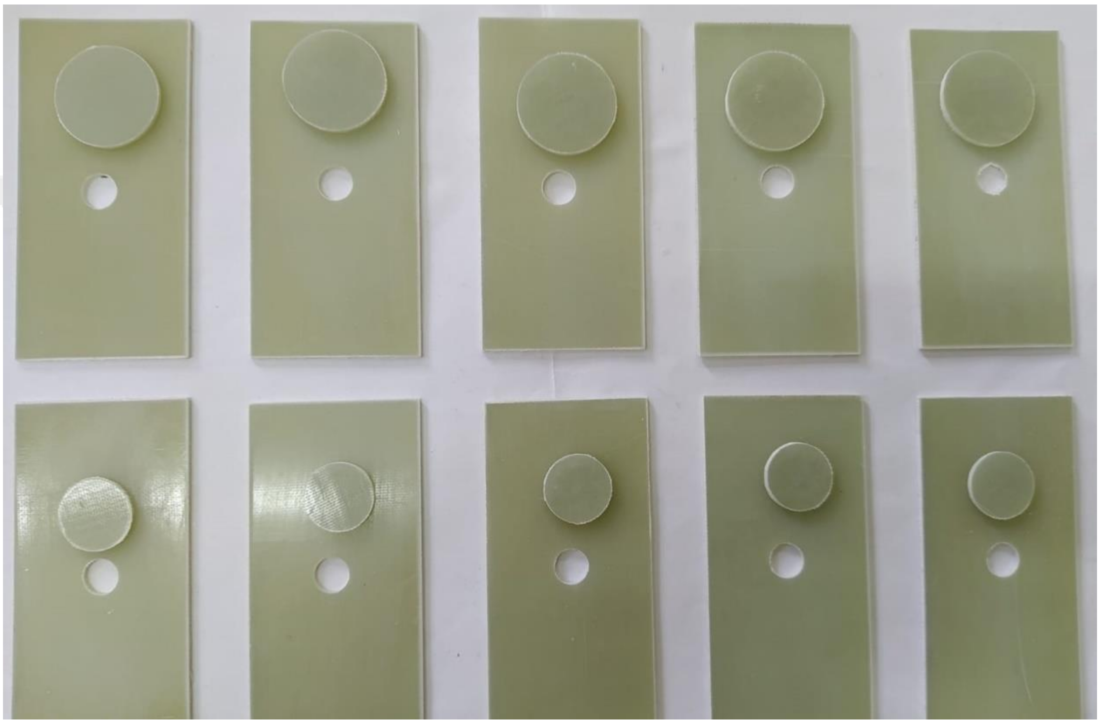

**Figure 3.7** The composite plate which is cut and drilled.

Secondly, mechanical abrasion is needed to smooth surfaces of composite plates before the adhesive bonding process. Sandpaper was used to smooth the surface of the composite plates and patches. Next, the surface of the composite plates was cleaned with acetone. Abrasive paper was used to prepare the bonding areas before the adhesive bonding. It's shown Figure 3.1.

Thirdly, preparing epoxy resin. three types of nanoparticles were used in this experiment, and these are nano-silica, nano-clay, and nano-graphene. Nanoparticles, hardener, and epoxy resin were stirred in different ratios and these proportions are respectively, nano-silica particles were added by 0,1, 2, 3, 5, and 10 % wt., nano-clay particles were added by 0, 1, 2, 3, 4, and 5% wt. and nano-graphene particles were added by 0, 0.05, 0.1, 1, 3, and 5 %wt. the mixing ratio of nano-silica, nano-clay, and nano-graphene. Nanoparticles, hardener, and epoxy resin were stirred in different ratios at 12000 rpm for 5 minutes by the mixer. It is shown in Figure 3.8.

**Figure 3.8** Nanoparticles, hardener, and epoxy resin.

### 3.4 Bonding of Plates and Patches with Prepared Epoxy Resin

In this study, single strap repair (SSR) has been chosen to bonding plates and patches. Plates and patches were bonded with a mixture of nano-silica, hardener, and epoxy resin. Five samples were used for each test with patch ratios are D/d= 2 and D/d=3.

The prepared epoxy resin was applied to the surfaces of the patches and around the holes of the plates. Then plates and patches were bonded to each other. It is shown in Figure 3.9.

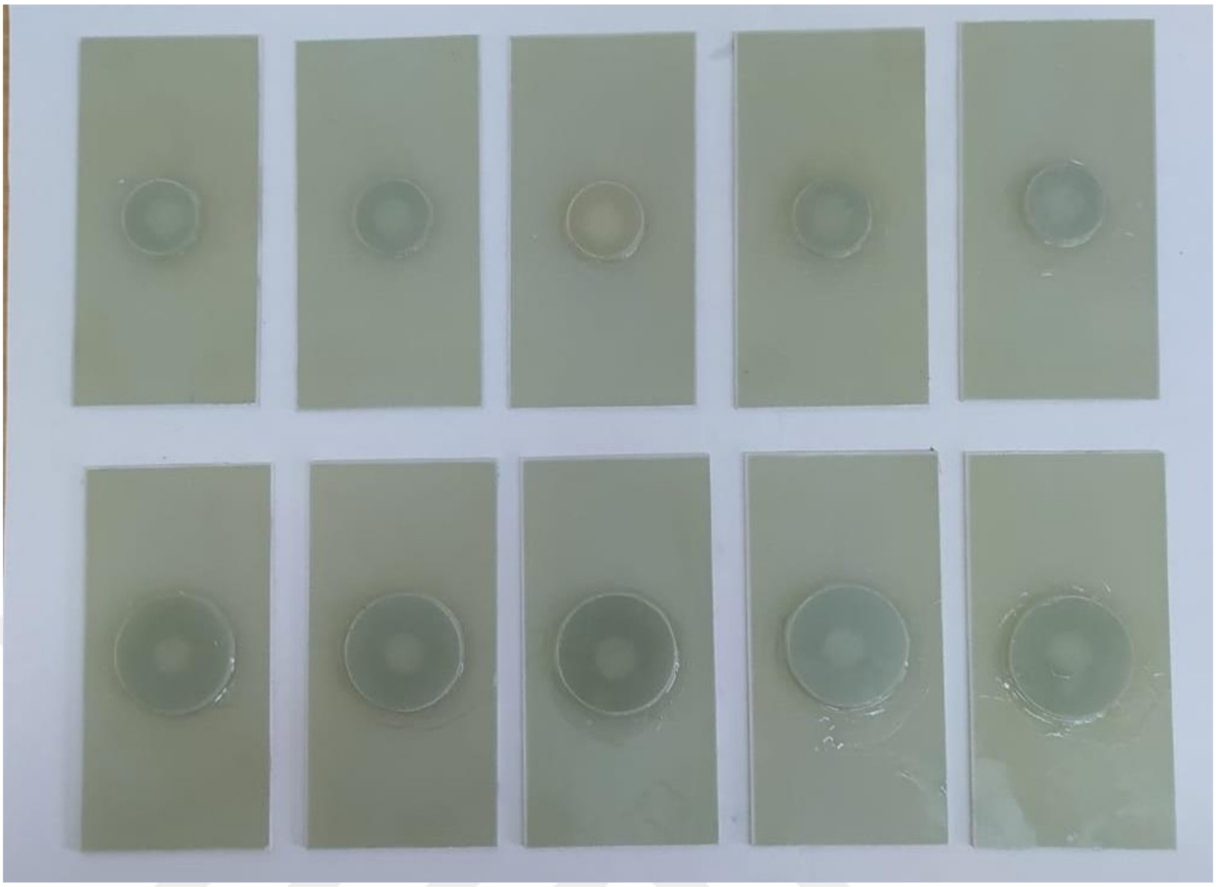

**Figure 3.9** Bonded plates and patches.

## 3.5 Experimental Tests

### 3.5.1 Tensile Test

Tensile tests were achieved on the Shimadzu AG-X controlled tensile test machine. The machine worked with 300 kN at a crosshead speed of 1 mm. min $^{-1}$. Shimadzu AG-X machine is viewed in figure 3.1. This machine has two heads that move relative to each other. The lower head is fixed while the upper head moves with a selected constant speed. Experiment plates connected to the jaws of the tensile machine by gripping areas of 50 × 10 mm. It is shown in Figure 3.10.


**Figure 3.10** Experiment plates connected to the jaws of the tensile machine.

The loading direction was vertically oriented before testing, similar to the tensile test. Samples were pulled at a cross head speed of 1.0 mm/min before failure. Trapezium software mounted in a computer (control unit) attached to the measuring system performed control, data collection, and processing. The force values and corresponding displacements were also reported by means of the test device control unit. In deciding stress-strain curves for the adhesive joints, these values are used. This arrangement ensured that no slip. For each group, five reiterated test examples were used and their average value of test of for each group results were taken.

# CHAPTER 4

# RESULTS AND DISCUSSIONS

In this chapter, the consequences of the tensile tests which are conducted to the determination of the mechanical features of the prepared single strap repair are shown.

## 4.1 Effect of Nano-Silica Particles

Single strap repair of samples was prepared with nano-silica in epoxy composite adhesives and tested under tensile test. The repair based on different weight ratios of nano-silica particles (0, 1, 2, 3, 5 and 10 % wt.) were tested and compared with control samples of pure epoxy.

Two separate patch repair ratios (D/d = 2 and D/d= 3) evaluated the patch repair output of the samples. By combining adhesive epoxy, tensile effects were tested for six separate mass ratios of nano silica particles (0, 1, 2, 3, 5 and 10 percent). The outcome of the nano-silica particles of tensile experiments are listed in Table 4.1.

**Table 4.1** Tensile test results of nano-silica particles.

| **Sample (D/d=2)** | **Fmax (kN)** | **Displacement at break (mm)** | **Sample (D/d=3)** | **Fmax (kN)** | **Displacement at break (mm)** |
|---|---|---|---|---|---|
| Without hole | 33.61 | 6.06 | Without hole | 33.61 | 6.06 |
| NS-0 | 17.90 | 3.63 | NS-0 | 17.60 | 3.94 |
| NS-1 | 16.87 | 3.15 | NS-1 | 17.10 | 3.20 |
| NS-2 | 16.98 | 3.39 | NS-2 | 17.64 | 3.42 |
| NS-3 | 17.39 | 3.40 | NS-3 | 18.18 | 3.59 |
| NS-5 | 15.54 | 3.48 | NS-5 | 16.89 | 4.85 |
| NS-10 | 14.17 | 3.75 | NS-10 | 16.36 | 4.42 |

The relationships of force-deformation and their maximum average values are shown in Figures 4.1 and 4.2. As can be seen from the result, samples which the patch ratio are D/d = 2, the amount of nano silica in epoxy resin up to 3% increased the tensile strength value of the samples but after the amount of nano silica is greater than 3% the tensile strength value of the samples were decreased. The tensile strength value of the samples is lower than those the without silica. The amount of nano silica in the epoxy resin with patch ratio D/d = 3 increased the tensile strength value of the samples up to 3%, but after the nano silica amount was above 3%, the tensile strength value of the samples decreased, just like the samples with the patch ratio D/d = 2. Unlike the patch ratio D/d = 2, in samples with patch ratio D/d = 3, the tensile strength of the amount of nano silica in epoxy resin added up to 3% is higher than without the silica in epoxy resin.

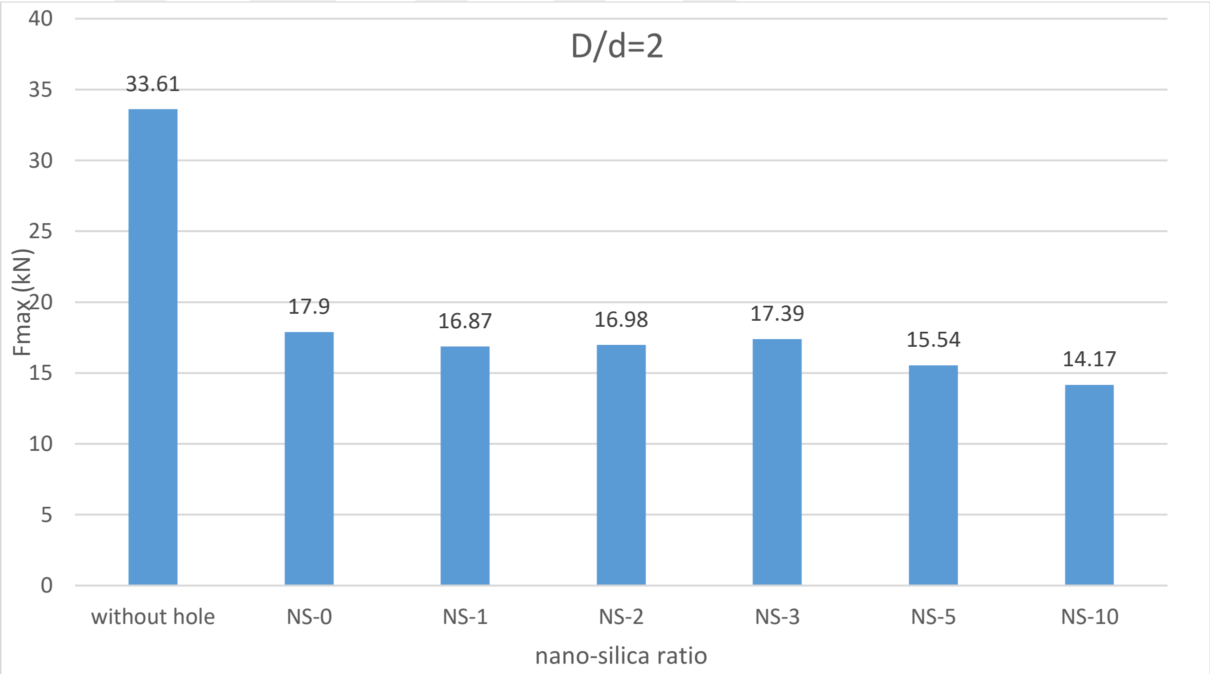


**Figure 4.1(a)** Maximum tensile force values for D/d=2 ratio.

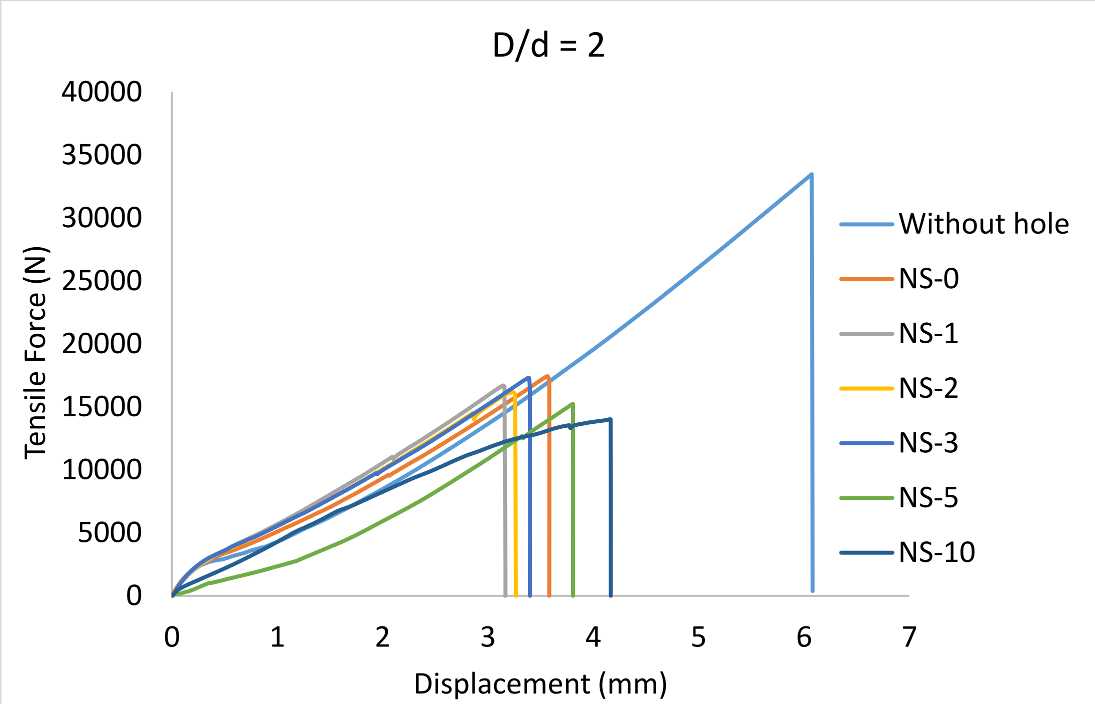


**Figure 4.1(b)** Tensile test load-displacement curves of samples for D/d=2 ratio.

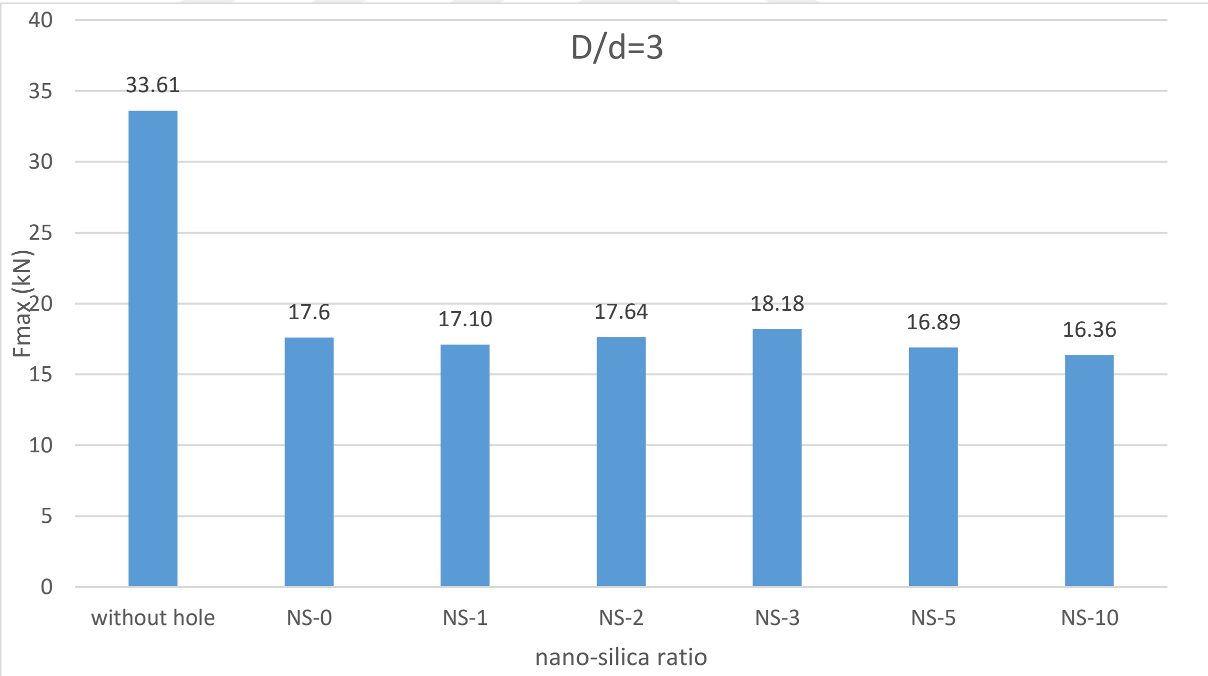


**Figure 4.2(a)** Maximum tensile force values for D/d=3 ratio.

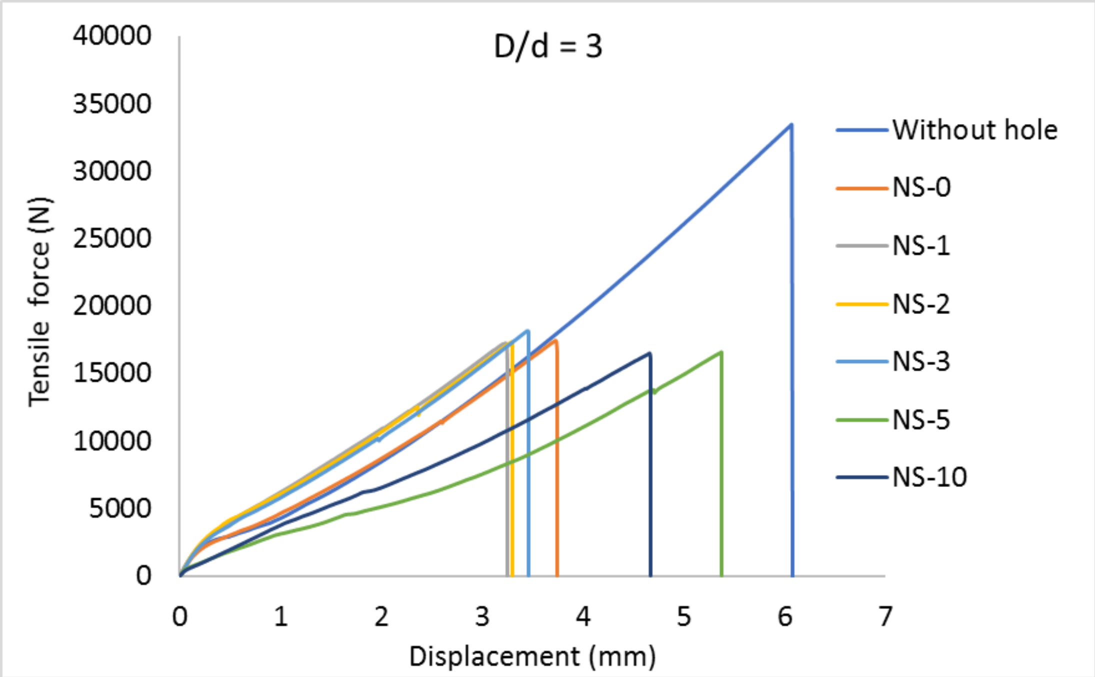


**Figure 4.2(b)** Tensile test load-displacement curves of samples for D/d=3 ratio.

The particles remaining on the plates after the test are more visible when the patch diameter is large. This was also ascribed to the distribution and weight percentages of particles in the epoxy, resulting in the higher strength between particle-matrix interfaces (shown in Figure 4.3). Opening a middle hole, however, resulted in a tensile strength decrease of around 47.61 percent.

As it is understood from the results, the effect of nano-silica particles increases as the patch diameter increases. Maximum tensile strength was recorded at 3% wt. content of nano-silica. After this ratio the tensile strength has decreased in consequence of bad dispersion between epoxy and nano-silica particles. According to test results, the overall tensile strength of single strap repair output (3 percent wt. nano-silica particle content) was reported as 3.29 percent for D/d = 3 compared to unmixed epoxy adhesive.

Some broken samples after tensile tests are shown in figure 4.3. It can be observed that the damaged surfaces of the composites repaired with epoxy adhesive having nano-silica particles are affected by the proportion of the filler material. The increase in the ratio of nano-silica materials in the epoxy caused an increase in the brittle behavior of the adhesive and a decrease in its load carrying capacity.

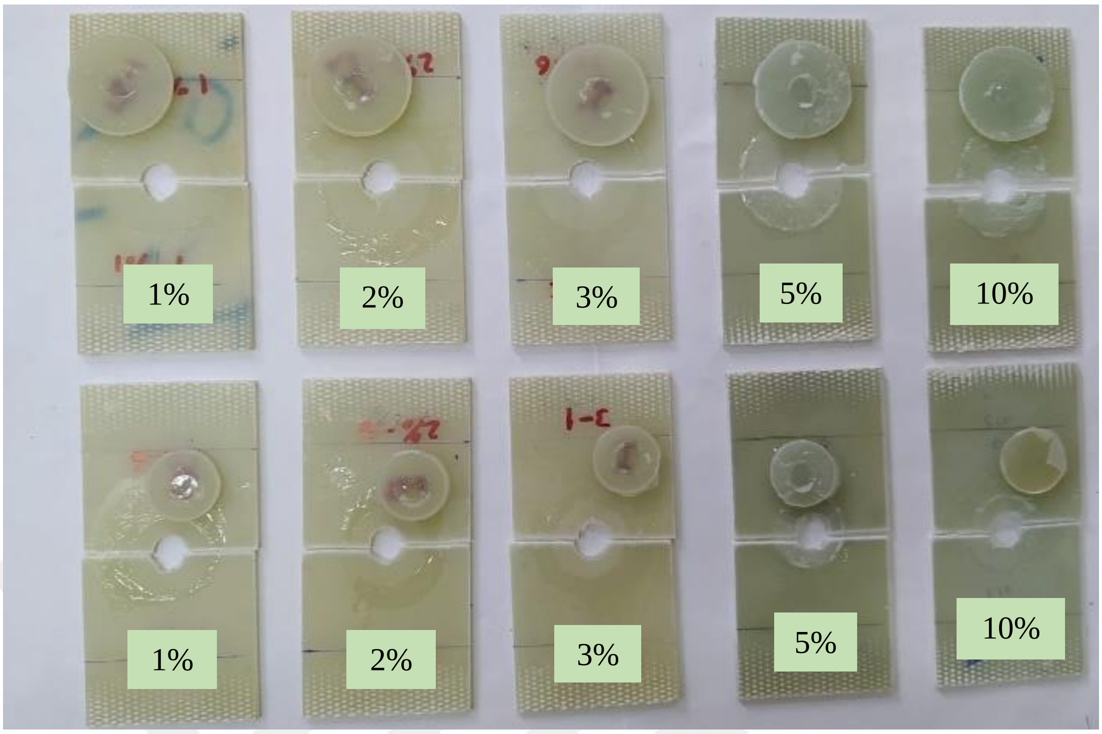


**Figure 4.3** After tensile tests broken samples repaired with nano-silica added epoxy adhesive.

## 4.2 Effect of Nano-Clay Particles

Single strap repair of samples which prepared were tested with different nano-clay content ratios 1wt. %, 2wt. %, 3wt. %, 4wt. %, and 5wt. % in epoxy composite adhesives. Nano-clay contents were used in production and tested under tensile test. It's compared the samples of pure epoxy.

Two separate patch repair ratios (D/d = 2 and D/d= 3) evaluated the patch repair output of the samples. By combining adhesive epoxy, tensile effects were tested for six separate mass ratios of nano clay particles (0, 1, 2, 3, 4 and 5 percent). The outcome of the nano-clay particles of tensile experiments are listed in Table 4.2.

According to the tensile test results which are patch ratio D/d = 2, the sample with a weight ratio of 3% in different weight ratio of nano-clay particles in epoxy resin gave a higher value than pure epoxy resin. After this ratio tensile strength has decreased. All samples which are patch ratio D / d = 3 have higher tensile test values than pure epoxy. But still after the 3% weight ratio of nano-clay in epoxy resin the tensile strength decreased slightly. As in nano- silica particles doped epoxy resin, in experiments with nano-clay particles doped epoxy resin samples with patch ratios D/d=3 gave better results than examples with patch ratio D/d=2. The relationships of force-deformation and their maximum average values are seen in Figures 4.4 and 4.5. The tensile test results showed that NCP was found to be the most effective particles among the three nano-particles in single strap repair.

**Table 4.2** Tensile test results of nano-clay particles.

| **Sample (D/d=2)** | **Fmax (kN)** | **Displacement at break (mm)** | **Sample (D/d=3)** | **Fmax (kN)** | **Displacement at break (mm)** |
|---|---|---|---|---|---|
| Without hole | 33.61 | 6.06 | Without hole | 33.61 | 6.06 |
| NC-0 | 17.90 | 3.63 | NC-0 | 17.60 | 3.94 |
| NC-1 | 15.54 | 3.42 | NC-1 | 19.80 | 3.58 |
| NC-2 | 14.36 | 2.72 | NC-2 | 18.32 | 3.35 |
| NC-3 | 18.40 | 4.52 | NC-3 | 21.04 | 5.07 |
| NC-4 | 16.93 | 3.59 | NC-4 | 21 | 3.86 |
| NC-5 | 15.51 | 3.60 | NC-5 | 19.13 | 4.99 |

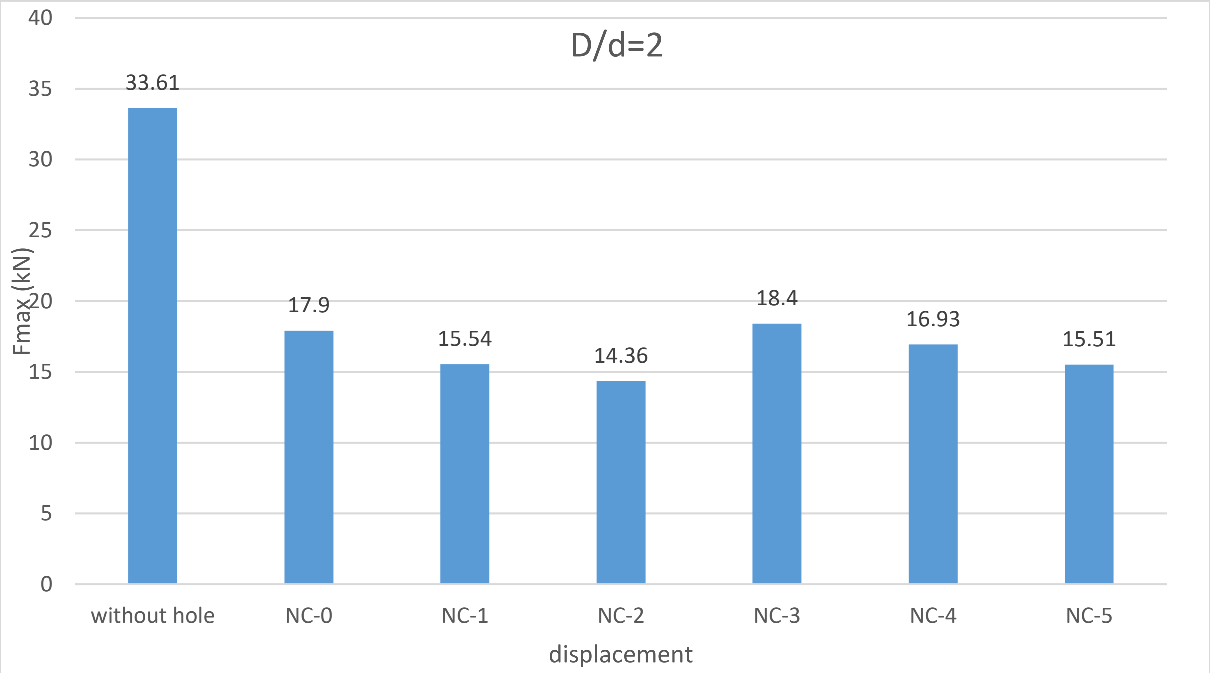


**Figure 4.4(a)** Maximum tensile force values for D/d=2 ratio.

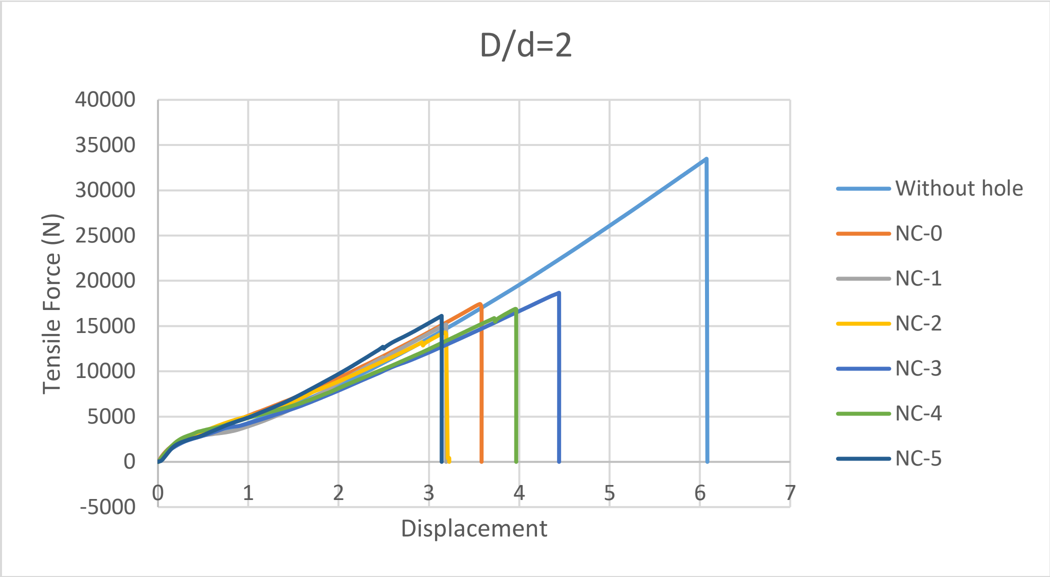


**Figure 4.4(b)** Tensile test load-displacement curves of samples for D/d=2 ratio

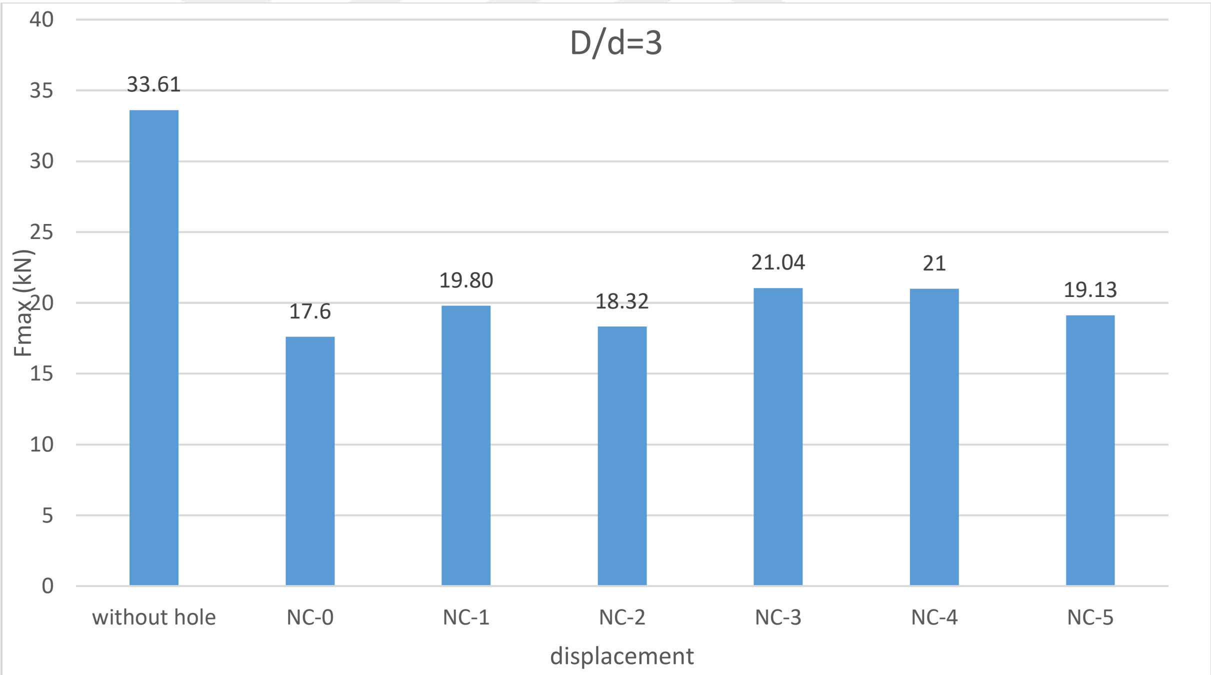


**Figure 4.5(a)** Maximum tensile force values for D/d=3 ratio.

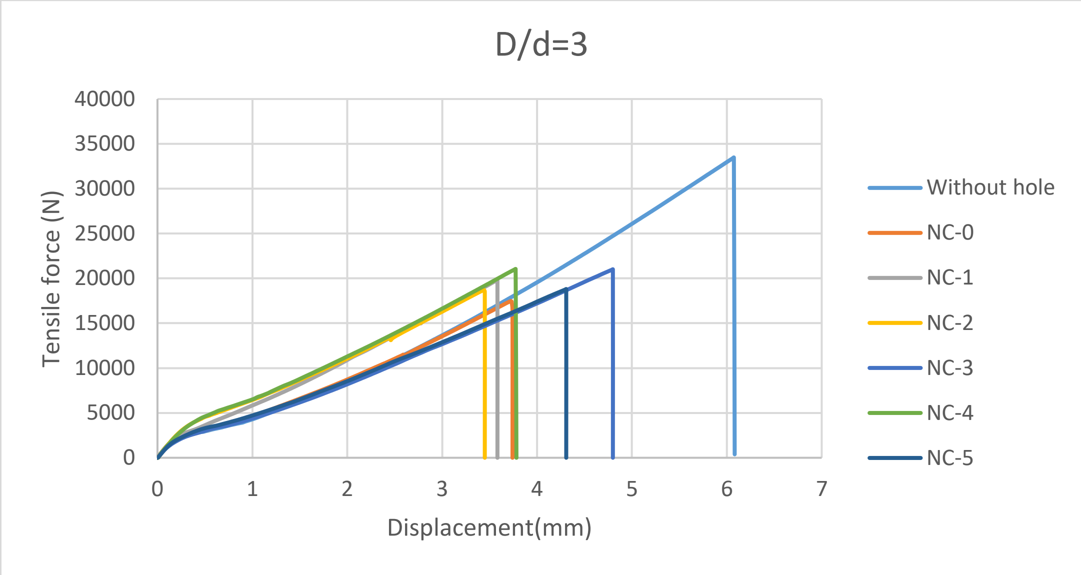


**Figure 4.5(b)** Tensile test load-displacement curves of samples for D/d=3 ratio.

As can be seen clearly from the results, the effect of nano-clay particles increases as the patch diameter increases. Maximum tensile strength was recorded at 3% wt. content of nano-clay. Compared with the unmixed epoxy adhesive, the maximum single strap repair output in tensile strength (3% wt. content of nano-clay particles) was reported as 16.34 percent for D/d = 3.

Some broken samples after tensile tests are shown in Figure 4.6. It can be observed that the damaged surfaces of the composites repaired with epoxy adhesive having nano-clay particles are affected by the proportion of the filler material. The increase in the ratio of nano-clay materials in the epoxy caused an increase in the brittle behavior of the adhesive and a decrease in its load carrying capacity.

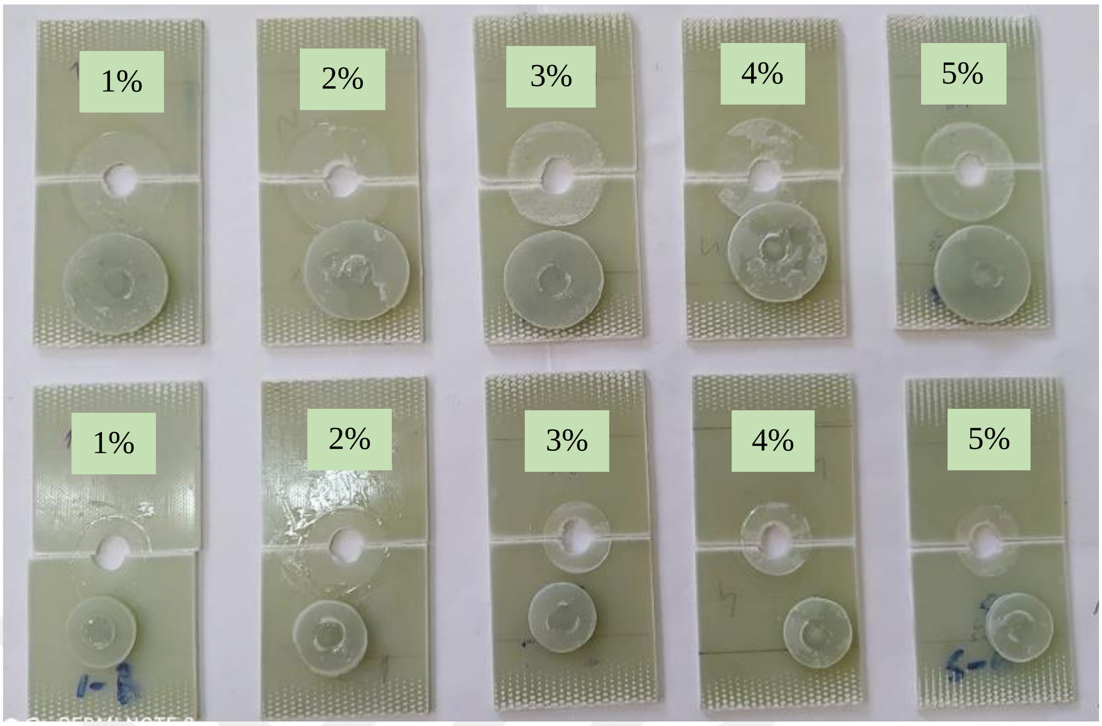


**Figure 4.6** After tensile tests broken samples repaired with nano-clay added epoxy adhesive.

### 4.3 Effect of Nano-Graphene Particles

In addition to nano-clay particles and nano-silica particles, nano-graphene particles were applied to reinforce epoxy and prepare single strap repair. These repairs were tested by tensile test. The results of the tensile testing of pure and nano particles strengthened epoxy were analyzed and compared with each other.

Two distinct patch repair ratios (D/d = 2 and D/d = 3) were examined for patch repair efficiency of the examples. By combining adhesive epoxy, tensile effects were tested for six distinct mass ratios of nano-graphene particles (0, 0.05, 0.1, 1, 3, and 5 % wt.), just like in the nano-silica and nano-clay experiment. The results of the tensile tests of nano-graphene particles are listed in Table 4.3.

All tensile test result of nano-graphene particles (NGP) which are patch ratio D/d = 2 and D/d=3 have lower tensile test values than pure epoxy. Samples which are patch D/d=2, the best recorded tensile test result of nano-graphene particles is 1% by weight ratio. After this ratio tensile tests result tend to decrease. Also, the tensile test results of NGP in epoxy resin with a weight ratio of 0.05% are greater than the tensile test results of nano-graphene particles in epoxy resin with a weight ratio of 0.1% which are patch ratio D/d = 2. Samples which are patch D/d=3, the best recorded tensile test result of nano-graphene particles is 0.1% by weight ratio and its 17.52 kN and after this ratio tensile tests result tend to decrease. The relationships of force-deformation and their maximum average values are seen in Figures 4.7 and 4.8. Tensile test results showed that NGP was the least effective particle among of the three nano particles in a single strap repair.

**Table 4.3** Tensile test results of nano-graphene particles.

| Sample (D/d=2) | Fmax (kN) | Displacement at break (mm) | Sample (D/d=3) | Fmax (kN) | Displacement at break (mm) |
|---|---|---|---|---|---|
| Without hole | 33.61 | 6.06 | Without hole | 33.61 | 6.06 |
| NG-0 | 17.90 | 3.63 | NG-0 | 17.60 | 3.94 |
| NG-0.05 | 14.79 | 2.64 | NG-0.05 | 15.66 | 2.50 |
| NG-0.1 | 14.27 | 2.53 | NG-0.1 | 17.52 | 3.88 |
| NG-1 | 16.02 | 3.18 | NG-1 | 16.84 | 2.68 |
| NG-3 | 15.68 | 3.34 | NG-3 | 15.92 | 3.06 |
| NG-5 | 15.34 | 3.63 | NG-5 | 15.85 | 3.53 |

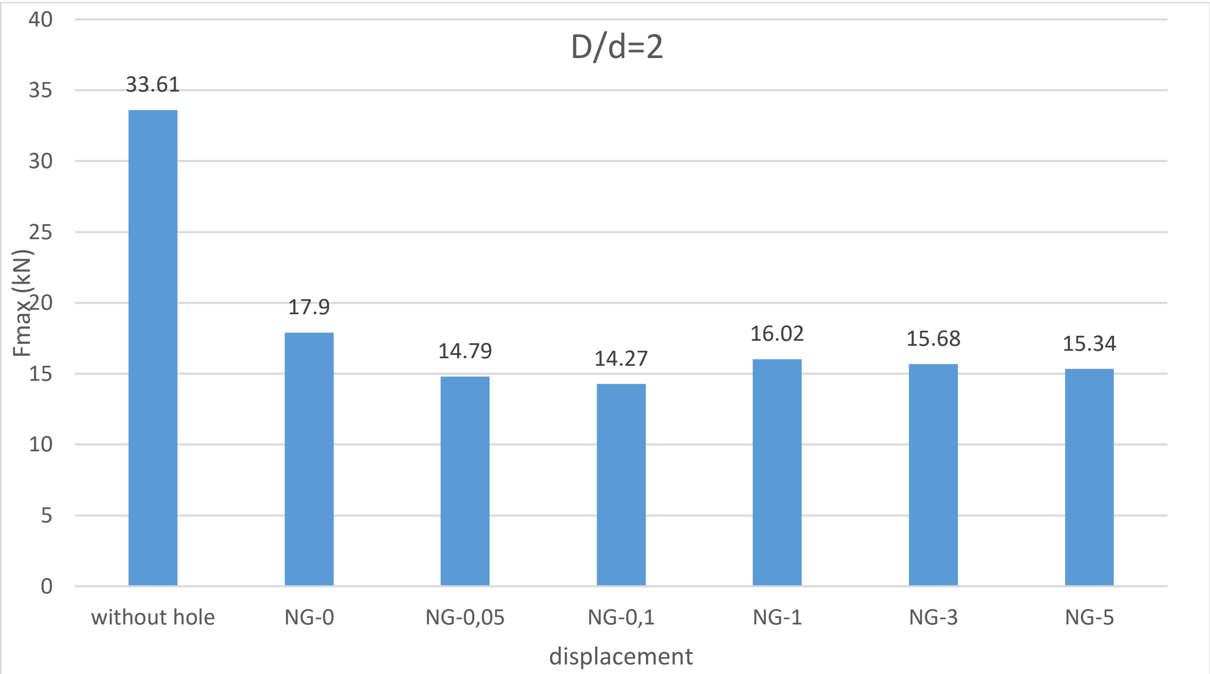

**Figure 4.7(a)** Maximum tensile force values for D/d=2 ratio.

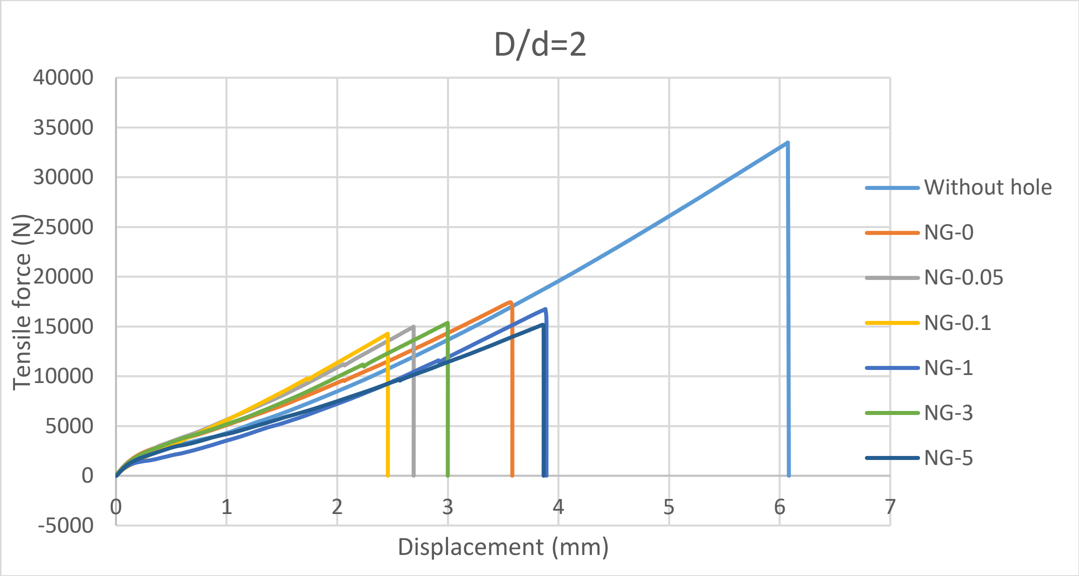


**Figure 4.7(b)** Tensile test load-displacement curves of samples for D/d=2 ratio

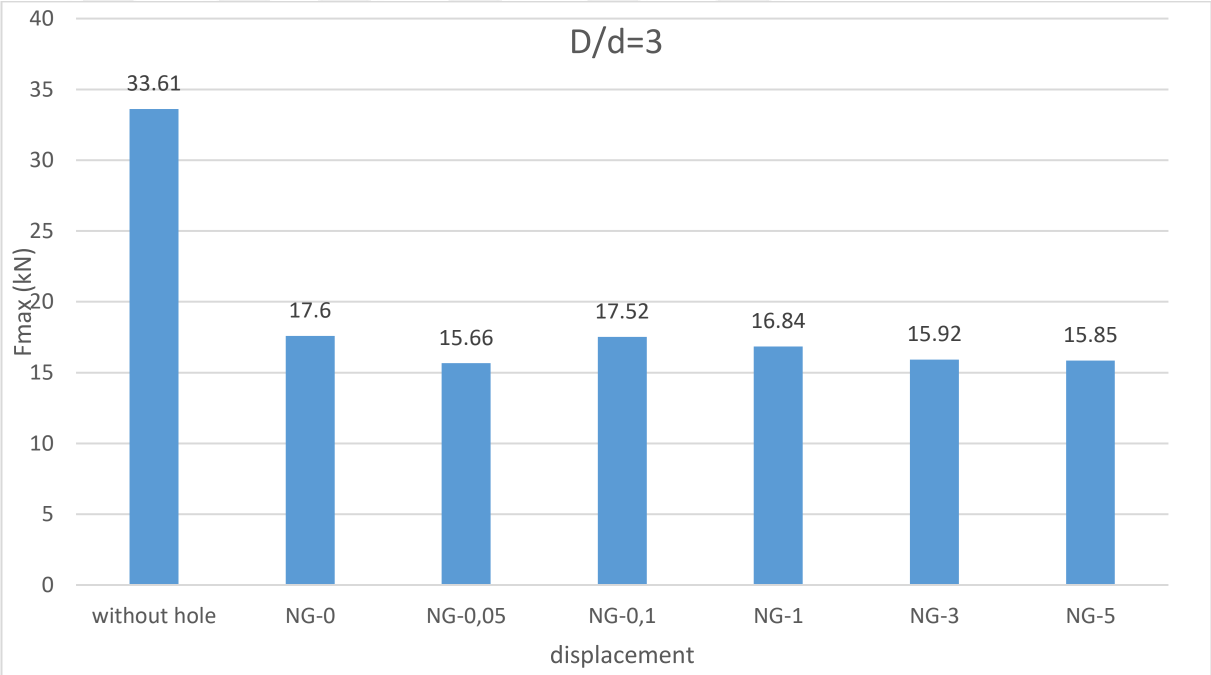


**Figure 4.8(a)** Maximum tensile force values for D/d=3 ratio.

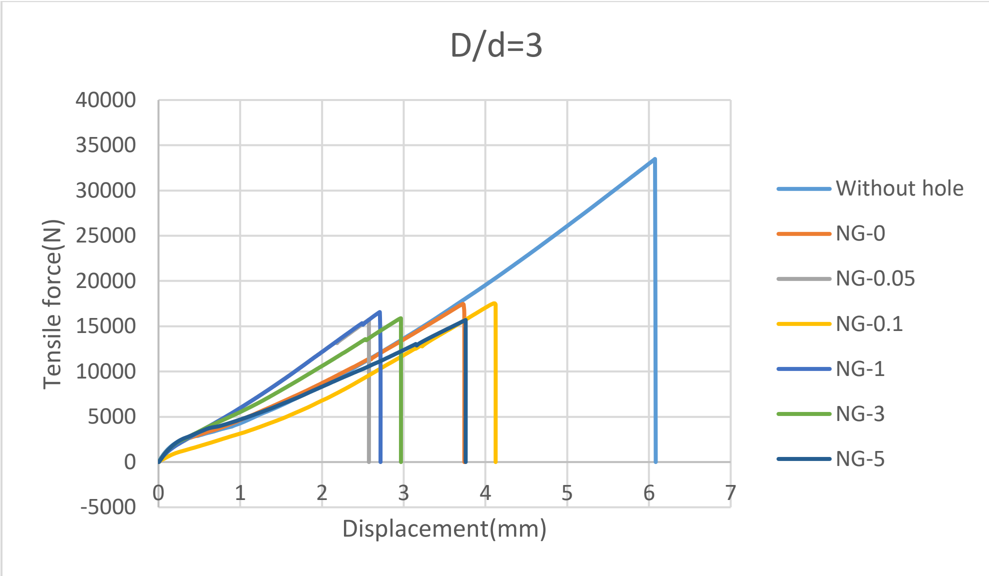


**Figure 4.8(b)** Tensile test load-displacement curves of samples for D/d=3 ratio.

According to the results, the effect of nano-graphene particles decreases tensile strength value of single strap repair. Maximum tensile strength was recorded at 0.1% wt. content of nano-graphene. Compared to the unmixed epoxy adhesive, the highest single strap repair output in tensile strength (0.1 percent wt. content of nano-graphene particles) was reported as -0.45 percent for D/d= 3.

Some broken samples after tensile tests are shown in Figure 4.9. It can be observed that the damaged surfaces of the composites repaired with epoxy adhesive having nano-graphene particles are affected by the proportion of the filler material. The increase in the ratio of nano-graphene materials in the epoxy caused an increase in the brittle behavior of the adhesive and a decrease in its load carrying capacity.

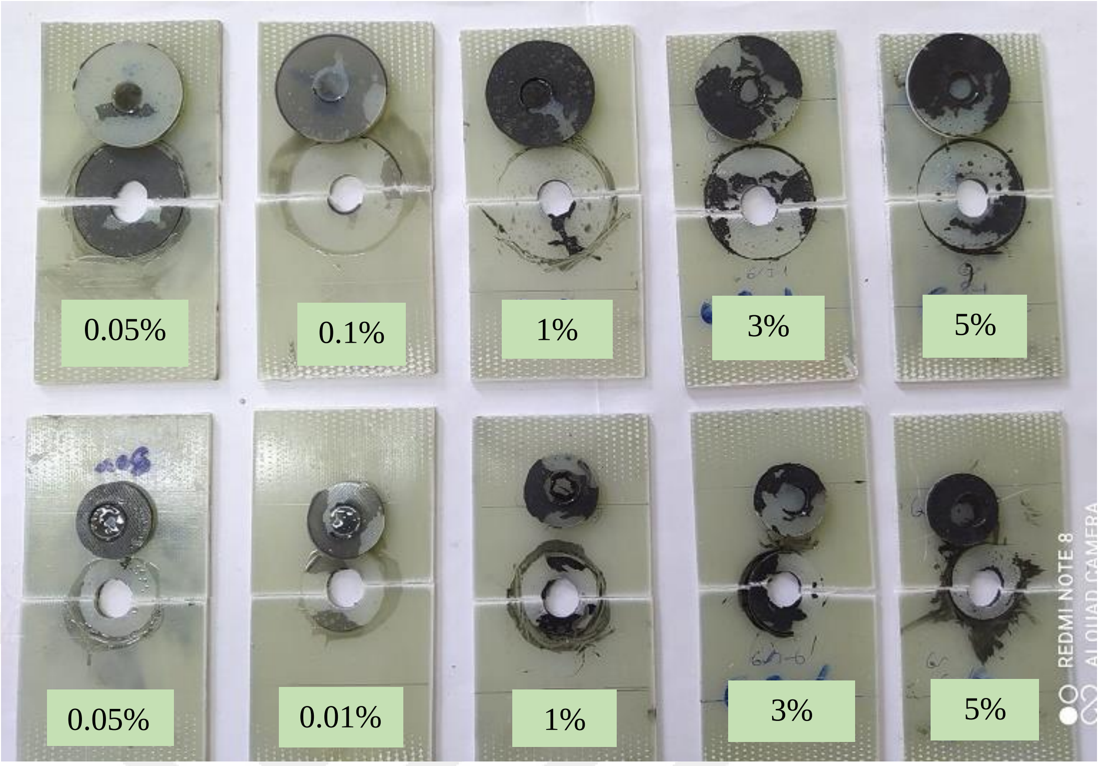


**Figure 4.9** After tensile tests broken samples repaired with nano-graphene added epoxy adhesive.

Among the nanoparticles added to the epoxy at different rates in the experiments, the tests that yield the best results are as follows, respectively. 3 % wt. nano-silica particle content was reported as 18.18 kN, 3% wt. content of nano-clay particles was reported as 21.04 kN, 0.1 percent wt. content of nano-graphene particles was reported as 17.52 kN with patch ratio is 3 and , the tests that yield the best results are as follows, respectively. 3 % wt. nano-silica particle content was reported as 17.39 kN, 3% wt. content of nano-clay particles was reported as 18.4 kN, 1 percent wt. content of nano-graphene particles was reported as 16.02 kN with patch ratio is 3 The maximum results of the tensile tests of nano-particles particles are listed in Table 4.4 and it's shown Figure 4.10.

**Table 4.4** The maximum results of the tensile tests of nano-particles particles.

| Sample D/d=2 | Fmax (kN) | Sample D/d=3 | Fmax (kN) |
|---|---|---|---|
| NS-3 | 17.39 | NS-3 | 18.18 |
| NC-3 | 18.40 | NC-3 | 21.04 |
| NG-1 | 16.02 | NG-0.1 | 17.52 |

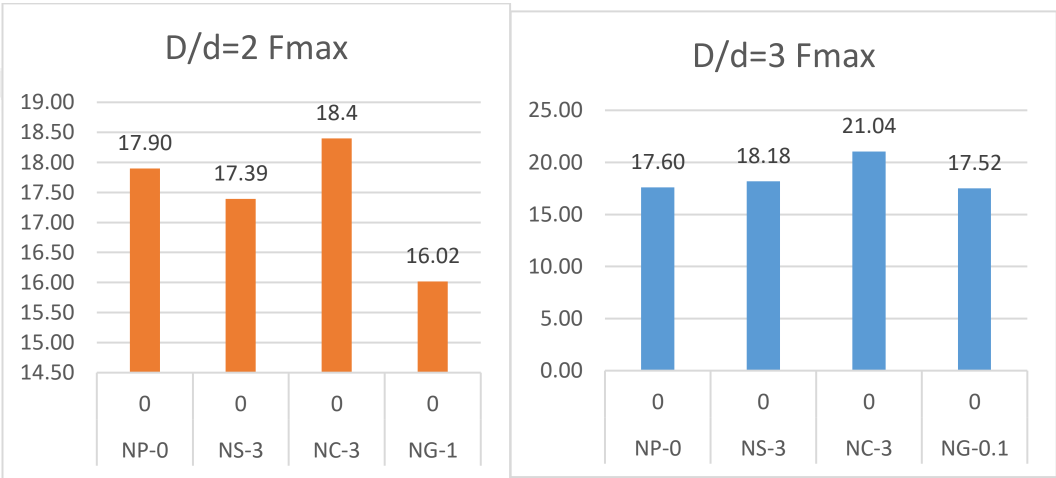


**Figure 4.10** The maximum results of the tensile tests of nano-particles.

Bulut et al. [36] studied with the use of micro-scale perlite and sewage sludge ash (SSA) fragments for glass-epoxy laminates, the tensile properties of epoxy adhesive include adhesively bound single-strap repairs. It was found the patch repairing performance of tensile test results of the experiments with D/d=3 is greater than samples with D/d=2. As can be seen in all the tests performed, the tensile test results of the experiments with a large patch ratio are higher than small patch ratio. The reason for this can be interpreted as follows; in experiments with a high patch ratio, the adhesion surface of nanoparticles increased, resulting in better resistance in the tensile test. Tutunchi et al. [44] have studied the effect of TiO2 nanoparticles on the adhesion strength of composite joints of steel-glass/epoxy bonded with two-part acrylic structural adhesives. The shear and tensile strengths of the adhesive joints have been shown to improve with the addition of up to 3 percent of the filler material, during which the addition of more filler content has reduced shear and tensile strengths of the adhesive connections. A decrease in the peel power of the connections was also caused by the incorporation of nanoparticles. Guadagno et al. [54] investigated the production of a new formulation of graphene nano-modified to increase the mechanical strength of structural adhesives. At a concentration of up to 1% wt., they find the epoxy adhesives loaded with graphene greatly improved the mechanical behavior of the bonded joints. The addition of 4 % wt. graphene has no major impact on mechanical efficiency only in the case of non-filled adherents. This is likely attributable to nanofiller agglomerations that induce heterogeneity at the interface between adherents and adhesives in large domains. The effect of the integration of graphene into the adherents, which has an effect on the chemical stability between adhesive and adherent surfaces, has contributed to a significant improvement in tensile strength relative to the corresponding unfilled adherent joints. This can be interpreted as follows; the efficiency of the single strap repair of composite laminates can be enhanced by combining nano particles with epoxy adhesive at an acceptable percentage of particles by weight. As the ratio of all nanoparticles rises, the adhesion of the epoxy has decreased. As can be seen from Tables 4.1, 4.2, and 4.3. As a result of the experiments, it will be the best option to use nano clay particles to rise the adhesion of epoxy on the plate. We can conclude that as the ratio of the nanoparticles in the epoxy increases, the adhesion of the epoxy decreased. The efficiency of the single strap repair of composite laminates can be enhanced by combining nano particles with epoxy adhesive at an acceptable percentage of particles by weight.

# CHAPTER 5

# CONCLUSION

In this study, effects of tensile features of epoxy adhesive with nano materials (nano-silica, nano-graphene, and nano-clay) on the glass epoxy laminates adhesively bonded single strap repairs are investigated. Result of the tensile tests are determined by using a 300 kN Shimadzu AG-X series universal testing machine. Based on the experimental results the following conclusions can be written from this study:

- Thanks to the enhanced load transition between particle-matrix interfaces, silica content at 3 percent wt., tensile strength achieves its optimum value. After this ratio the tensile strength has decreased in consequence of bad dispersion between epoxy and nano-silica particles.
- Compared with unmixed epoxy adhesives, the maximum single strap repair efficiency in tensile strength (3 percent wt. content of silica particles) was reported as 3.29 percent for D/d = 3.
- The patch repair efficiency of D/d = 3 samples is better than that of D/d = 2 samples.
- Maximum tensile strength was recorded at 3% wt. content of nano-clay. After the 3% weight ratio of nano-clay in epoxy resin the tensile strength decreased slightly.
- Compared to the pure epoxy adhesive, the maximum single strap repair efficiency in tensile strength (3 percent wt. content of nano-clay particles) was reported as 16.34 percent for D/d= 3.
- It has been found that all tensile test result of nano-graphene particles which are patch ratio D/d = 2 and D/d=3 have lower tensile test values than pure epoxy.
- Samples which are patch D/d=3, the best recorded tensile test result of nano-graphene particles is 0.1% by weight ratio and its 17.52 kN and after this ratio tensile tests result tend to decrease.

- Compared to the pure epoxy adhesive, the maximum single strap repair efficiency in tensile strength (0.1 percent wt. content of nano-graphene particles) was reported as -0.45 percent for D/d= 3.
- It was found that the effect of nano-graphene particles decreases tensile strength value of single strap repair.
- It has been observed that tensile test results showed that NGP was the least effective particle among of the three nano particles in a single strap repair.
- It has been found that the effect of nano-particles increases as the patch diameter increases.
- The particles remaining on the plates after the test are more visible when the patch diameter is large. This shows that the increase in the amount of silica added to the epoxy and the increase in patch diameter increases the tensile strength.
- It has been found that nanoparticles are useful for improving the mechanical features of composite adhesives.
- As a result, the repair efficiency of composite laminates can be increased by combining nano particles with epoxy adhesive at an acceptable particle weight percentage, helping to overcome a major engineering challenge during operation.

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