EFFECTS OF SURFACE TREATMENTS ON FATIGUE PERFORMANCE OF ADHESIVELY BONDED SINGLE LAP JOINT CARBON FIBER BASED POLYMER COMPOSITES

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by
Ahmet Ayberk GÜRBÜZ

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İZMİR

We approve the thesis of **Ahmet Ayberk GÜRBÜZ**

Examining Committee Members:

Prof. Dr. Metin TANOĞLU
Department of Mechanical Engineering, İzmir Institute of Technology

Prof. Dr. Engin AKTAŞ
Department of Civil Engineering, İzmir Institute of Technology

Assoc. Prof. Dr. Mehmet SARIKANAT
Department of Mechanical Engineering, Ege University

27 June 2024

Prof. Dr. Metin TANOĞLU
Supervisor, Department of Mechanical
Engineering, İzmir Institute of Technology

Prof. Dr. M. İ. Can DEDE
Head of the Department of Mechanical
Engineering

Prof. Dr. Mehtap EANES
Dean of the Graduate School

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ABSTRACT

EFFECTS OF SURFACE TREATMENTS ON FATIGUE PERFORMANCE OF ADHESIVELY BONDED SINGLE LAP JOINT CARBON FIBER BASED POLYMER COMPOSITES

In the period following the advent of new technologies, alternative joining techniques began to supplant traditional mechanical fasteners in applications involving carbon fiber-reinforced polymers. The majority of challenges associated with mechanical fasteners, including stress concentration, weight and the absorption of radar signals as well as corrosion, were effectively addressed by the introduction of adhesive bonding in the field of CFRPs. Nevertheless, several factors exert a significant influence on the adhesion strength, including the presence of contaminants and an excess of matrix on the surface layer.

The objective of this thesis is to examine the effects of applying surface treatment on the fatigue performance of adhesively bonded carbon fibre-reinforced polymer composite plates. Carbon fibre-reinforced polymer laminates with a stacking sequence of $[45/-45/45/0/-45/90]_s$ were manufactured using unidirectional prepregs by the autoclave technique. Two different surface treatments, namely laser treatment and electrospinning, were applied to the adhesion surface of the carbon fibre-reinforced polymer laminates. Load-controlled tension-tension fatigue tests were conducted to investigate the fatigue performance of composites subjected to different surface treatments. Specimens were subjected to cyclic loading at stress levels ranging from 30% to 50% of the average maximum single lap shear load, as determined from static single lap shear tests. The effects of surface treatments on the fatigue performance of the adhesion surface were interpreted using SEM images, stiffness degradation, and Wöhler curves.

ÖZET

YAPIŞKANLA BİRLEŞTİRİLMİŞ TEK BİNDİRMELİ KARBON FİBER ESASLI POLİMER KOMPOZİTLERİN YORULMA PERFORMANSINA YÜZEY İŞLEMLERİNİN ETKİLERİ

Yeni teknolojilerin ortaya çıkışını takip eden dönemde, alternatif birleştirme teknikleri karbon elyaf takviyeli polimerleri içeren uygulamalarda geleneksel mekanik bağlantı elemanlarının yerini almaya başlamıştır. Gerilme konsantrasyonu, ağırlık, radar sinyallerinin emilmesi ve korozyon gibi mekanik bağlantı elemanlarıyla ilgili zorlukların çoğu, CFRP'ler alanında yapıştırıcı bağların kullanılmaya başlanmasıyla etkili bir şekilde ele alınmıştır. Bununla birlikte, kirletici maddelerin varlığı ve yüzey tabakasındaki matris fazlalığı da dahil olmak üzere çeşitli faktörler yapışma gücü üzerinde önemli bir etkiye sahiptir.

Bu tezin amacı, yüzey işleminin uygulanmasının, yapışkan olarak bağlanmış karbon elyaf takviyeli polimer kompozit plakaların yorulma performansı üzerindeki etkilerini incelemektir. $[45/-45/45/0/-45/90]_s$ istifleme sırasına sahip karbon fiber takviyeli polimer laminatlar, otoklav tekniği ile tek yönlü prepregler kullanılarak üretilmiştir. Karbon fiber takviyeli polimer laminatların yapışma yüzeyine lazer işlemi ve elektrospinning olmak üzere iki farklı yüzey işlemi uygulanmıştır. Farklı yüzey işlemlerine tabi tutulan kompozitlerin yorulma performansını araştırmak için yük kontrollü çekme-germe yorulma testleri yapılmıştır. Numuneler, statik tek bindirmeli kesme testlerinden belirlenen ortalama maksimum tek bindirmeli kesme yükünün %30 ila %50'si arasında değişen gerilme seviyelerinde döngüsel yüklemeye tabi tutulmuştur. Yüzey işlemlerinin yapışma yüzeyinin yorulma performansı üzerindeki etkileri SEM görüntüleri, sertlik bozulması ve Wöhler eğrileri kullanılarak yorumlanmıştır.

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CHAPTER 1

INTRODUCTION

1.1. Definition of Composites

Composite materials consist at least two material which are different between them chemically and physically. These materials are classified into two main groups: the matrix phase and the reinforcement phase. The stronger side is the reinforcement phase. The reinforcement phase serves to strengthen the composite material and impart additional properties, such as heightened corrosion resistance, hardness, impact strength, heat resistance and fatigue strength.[1] A variety of fibres, including carbon, glass, aramid, nylon, and carbide, are employed in the industry. Carbon fibres are employed primarily in the aerospace industry, whereas glass fibres are utilised in the automotive industry. The matrix phase, which is typically composed of polymer, metal, or ceramic-based materials, serves a dual purpose: to surround the dispersed phase and to protect it from environmental or chemical reactions. Additionally, it serves to transfer the active load to the dispersed phase. Furthermore, these two phases are unable to react chemically with one another.[2]

Various properties of composite materials, such as their morphologies, degrees of crystallinity, components, proportions, and distributions, as well as the structure and composition of the interface, can be customised during the production of composite materials. This high level of customisation makes composite materials highly attractive in numerous industries, including automotive, construction, biomedical, aerospace and defence.[3]

The superior properties of fiber-reinforced polymer composite materials, such as high specific stiffness, high specific strength, and controlled anisotropy, make them a popular choice in many industries. In addition, properties of carbon fiber polymer matrix composites such as low density, high strength, good fatigue resistance, good creep resistance, high stiffness, low friction coefficient, toughness and damage tolerance are just some parts of properties for step forward to using it in aerospace industries. The diagram showing the areas where composite materials are used on the Boeing B787, one of the most well-known examples in aviation, is given in figure 1.1. Composite material consumption is 50% of the manufacturing process for the Boeing B787 as shown in figure 1.1.[1,3]

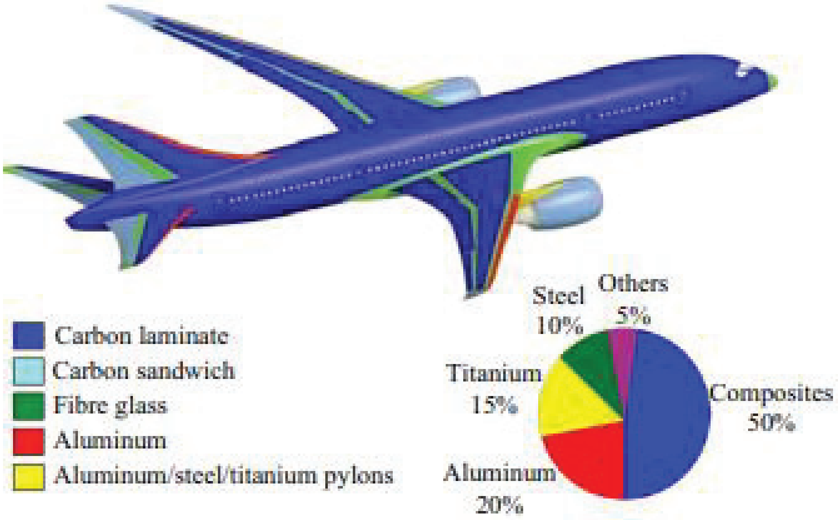

Figure 1.1. Application of composites in Boeing B787 (Source: Zhang, L., et al., 2019)

Fiber-reinforced polymer composite materials that are widely customisable than other materials are increasingly in demand not only in the aerospace industry, as previously mentioned, but also in the automotive, defence, marine, healthcare, transportation, construction, sport equipments, and energy sectors. Their use in these fields is expanding daily, with a growing number of applications. Additionally, the number of industries using these materials is increasing due to enhancements in their quality. Various applications of composite materials are represented in figure 1.2 below.

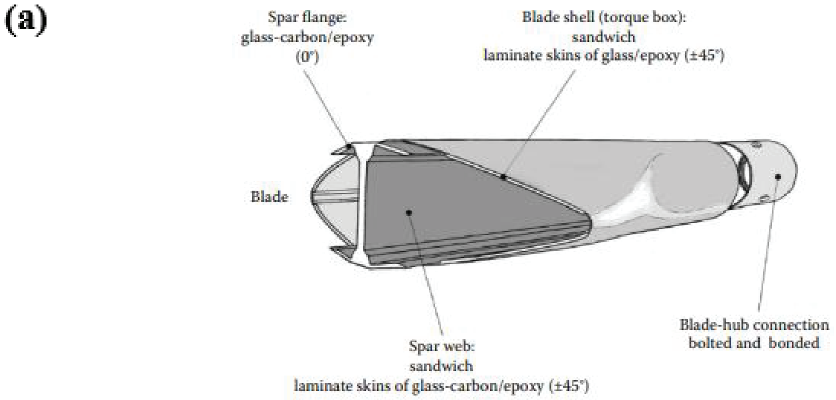

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Figure 1.2. Various applications of composite materials (a) wind turbine blade, (b) automotive, (c) surfboard, (d) marine (Source: Gay, D., et al., 2015)

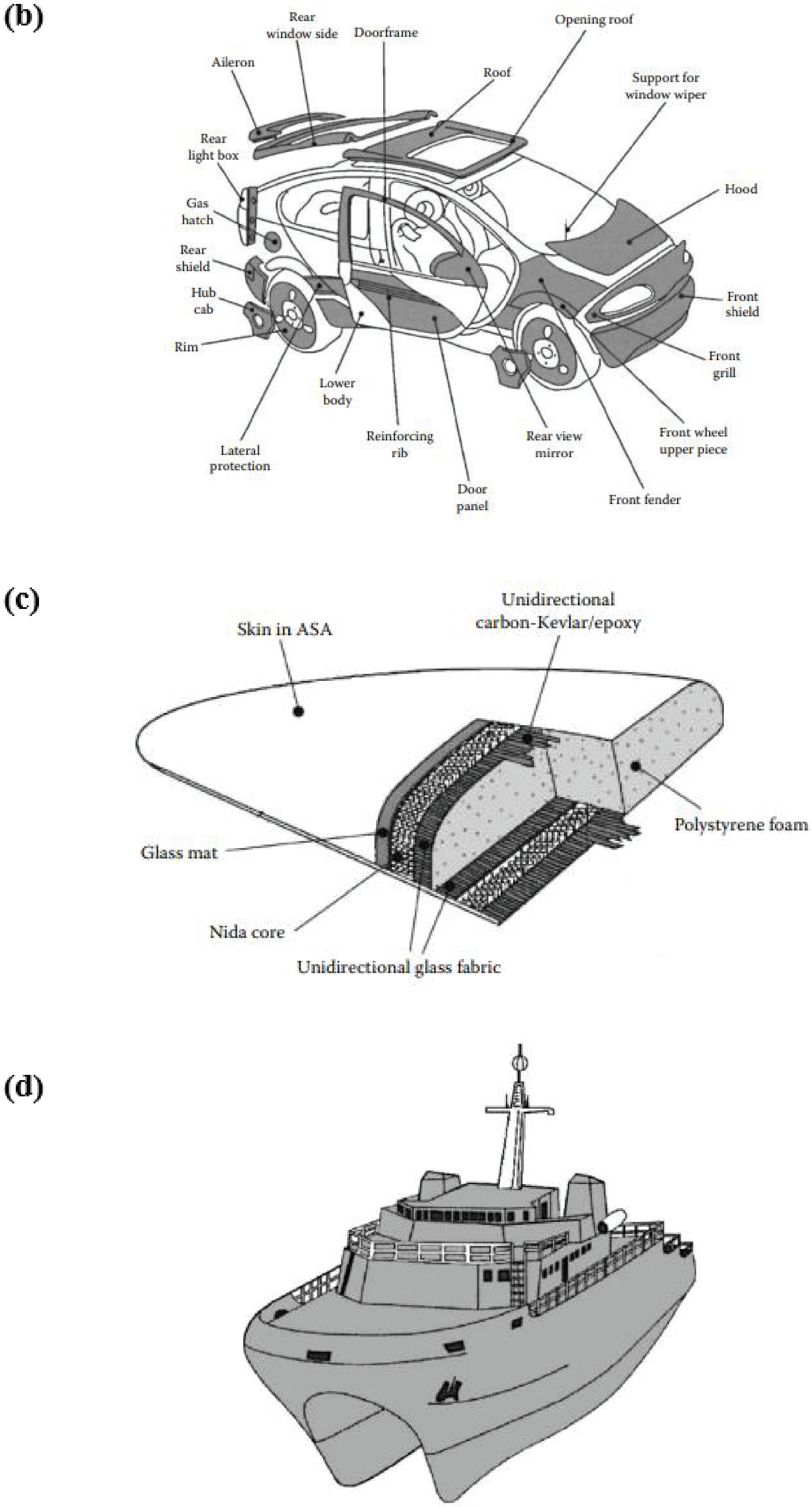

Figure 1.2. (cont.)

1.2. Classification of Composites

There are essentially two classification systems for composite materials. The first is predicated upon the type of matrix material, while the second is based upon the type of reinforcing material.[2]

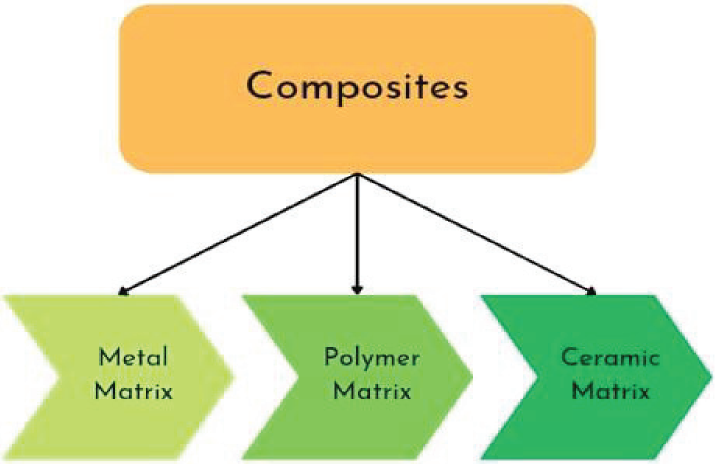

Figure 1.3. Classification of composites based on matrix material

Usually three different matrix materials, which are based on metallic, polymeric, and ceramic, can be used in composite materials, as shown in figure 1.3 above. The matrix material fulfils three main functions: firstly, it ensures the geometric arrangement of reinforcement materials; secondly, it protects reinforcement materials from environmental factors; and thirdly, it transmits loads to the reinforcement materials. The specific properties imparted to the composite depend on the type of matrix material used. To provide an example, it is preferable to use metal matrix composites for enhanced hardness and creep resistance in high temperatures. On the other hand, polymer matrix composites are preferred for increased stiffness, strength, environmental tolerance, toughness, unlimited shelf-life, and more advantages.[3,4]

To determine the behaviour of composite materials, the type of matrix material and the structure of the reinforcement material are both considered important. Figure 1.4 displays the categorisation of composites, based on the structure of the reinforcement material. These are classified into three main groups: particle-reinforced, fibre-reinforced, and structural composites.

Particle-reinforced composites can be classified into two types: large particle and dispersed-strengthened. The main difference between them is the particle size used for reinforcement. In addition, composites utilizing these reinforcement types enhance properties such as yield strength, tensile strength, and hardness.[2]

Fiber-reinforced composites can be classified into two groups which based on fiber length, as illustrated in figure 1.4. Fibers are the most important reinforcement phase in technologically. The design purpose of fiber-reinforced composites is usually to achieve high strength and stiffness on a weight basis. Fiber-reinforced composites prefer when high specific strengths and high specific moduli are aimed.[2]

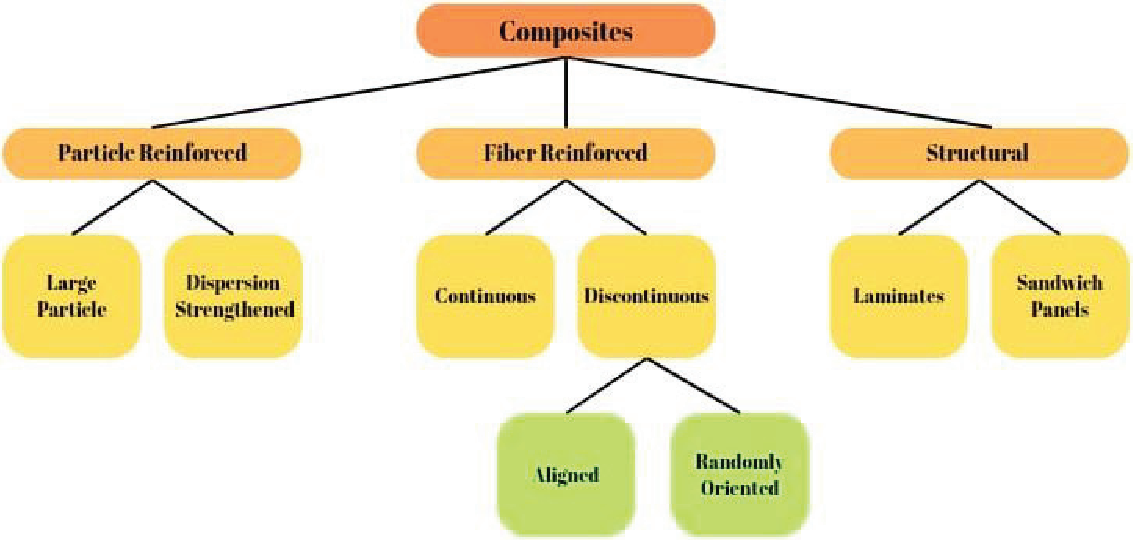

Figure 1.4. Classification of composites based on reinforcement material structure

Structural composites can be classified into two groups, which known as laminates and sandwich panels as illustrated in figure 1.4. Structural composites generally consist of multiple layers, and they have low density. In addition, structural composites are used when the applications require structural integrity, high tensile and compressive strength, torsional strength, and as well as high stiffness.[2]

1.3. Manufacturing Techniques for Prepreg Based Composites

Composite materials exhibit a number of exceptional properties, including a high degree of lightness, high stiffness and strength, ease of processing, and so forth. Composites are increasingly being employed in place of metals, ceramics, and woods, which are conventional materials due to their superior performance and excellent properties than conventional materials. In recent times, composites have been employed extensively, particularly within the aerospace industry. Figure 1.5 illustrates the constituent materials of composite materials and the manufacturing options available for them.[1,5,6]

The development of prepreg tape, which consists of precoated fibres with polymer resin, was a significant milestone in composite manufacturing technology. This development provides precise proportion between resin and fiber eliminating concerns regarding the proportion of fiber and resin needed in producing composite materials. In addition, the use of prepreg tape in autoclave moulding, which is a standard process in the aerospace industry for the production of composite materials, has become widespread due to the advantages it offers.[5]

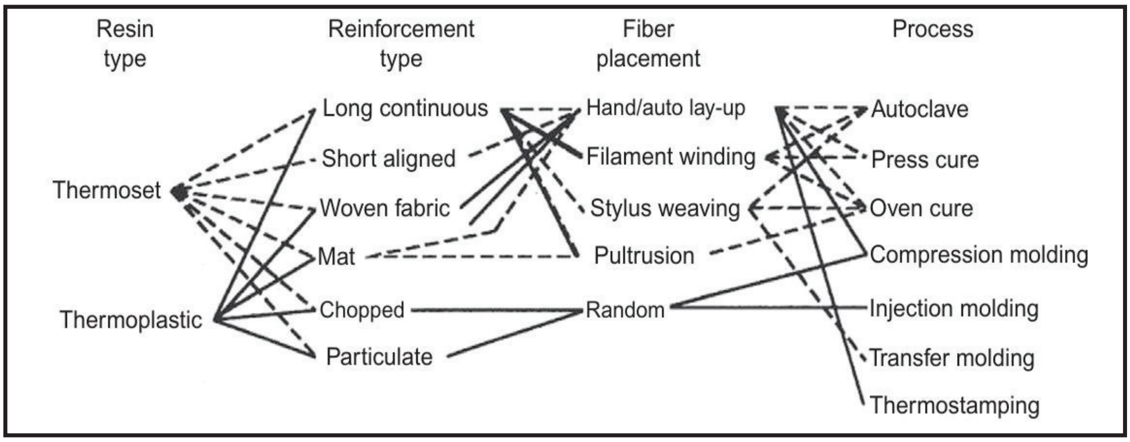

Figure 1.5. Constituent materials of composite and manufacturing options (Source: Balasubamanian, 2018)

The autoclave process is used to produce plates which has a high fiber volume fraction with high strength and stiffness. Because of this reason this manufacturing technique uses in aerospace and automotive industries where critical to using high strength and lightweight material.[1]

Autoclave technique generally follows the steps which is given below;

- Firstly, fabrics are prepared with using stacking sequences then laid up in a mold.
- Epoxy resin is applied the carbon fibers generally with resin transfer molding. On the other hand, in this thesis, prepregs (pre-impregnated with resin matrix) are used because of this reason this step just passed.
- That mold is placed into the autoclave.

- High pressure and temperature are applied into autoclave chamber to prepregs, these high pressure and temperature combination allows the epoxy to cure and bond with carbon fibers together.
- After curing process is completed carbon fiber reinforced plate is removed from mold and trimmed.

The autoclave process has some disadvantages as expensive, need specialized equipment, skilled personal to use it and high-quality material and time-consuming process compared to other manufacturing techniques of carbon fiber reinforced plates on the other hand higher quality plates (high strength and stiffness values, high fiber volume fraction etc.) producible with using this method as mentioned above. The schematic representation of autoclave technique is shown in figure 1.6.[5]

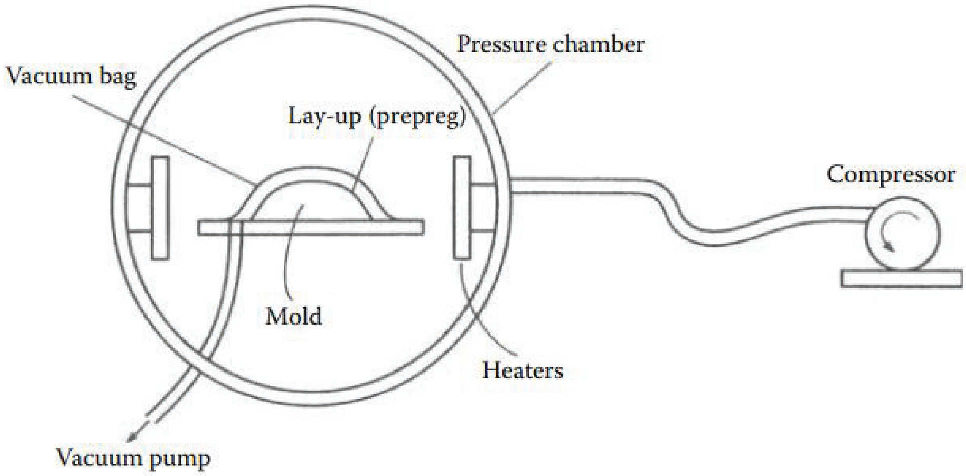

Figure 1.6. The schematic representation of autoclave technique (Source: Gibson, 2007)

1.4. Joining Methods for Composite Parts

As is well known, there are numerous methods of joining composite parts, including bonded joints, mechanical fasteners, hybrid joints, stitching, welding, and so forth. fIn addition to this, mechanical fasteners which also known as conventional joining method and bonded joints are used more than other methods.

Mechanical fasteners which are mostly bolt, and rivet joints are generally the first choice for joining because of they provide high joint strength and precision. In order to obtain all these good properties of mechanical joints need some requirements as like well quality of machined holes. When it comes to composite materials drilling process can be give some undesirable damages such as delamination and fiber pullout because of certain characteristics of composite parts which are anisotropic, non-homogenous and have hard reinforced fibers. And all these damages reduce the strength and performance of the materials. Two example for occurring delamination when are applied drilling process for mechanical joining on composite parts are given below in figure 1.7.[7]

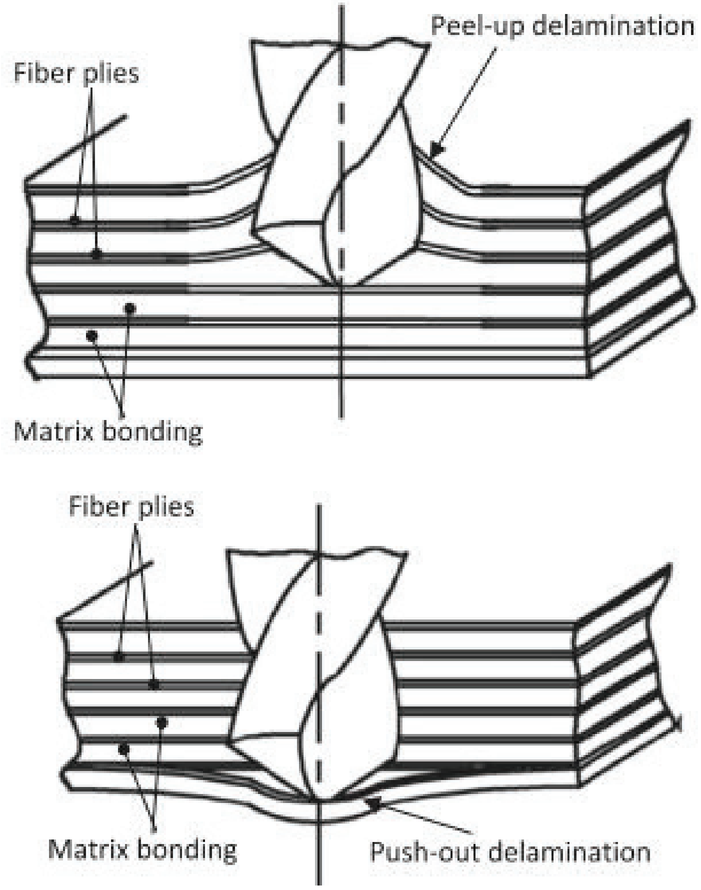

Figure 1.7. Occurring delamination results of apply drilling process
(Source: Liu, D. F., et al., 2012)

Nowadays, adhesive joints (bonded joints) are preferred instead of mechanical fasteners because of their superior properties and advantages such as providing similar or stronger mechanical properties, lower cost, light weight and the ability to transmit stresses from one part to another which join each other more uniformly than mechanical fasteners on composite materials.[8,9,10] However, bonded joints have mainly three different joining techniques which are known as co-curing, co-bonding, and secondary bonding. Briefly, co-curing refers to the combination of two uncured parts, co-bonding refers to one uncured part combined with another cured part for both co-curing and co-bonding techniques can be applied with or without adhesive. And finally secondary bonding contains to the combination of two cured parts with using a structural adhesive. These three joining techniques which are co-cuing, co-bonding and secondary bonding are illustrated in figure 1.8.[11]

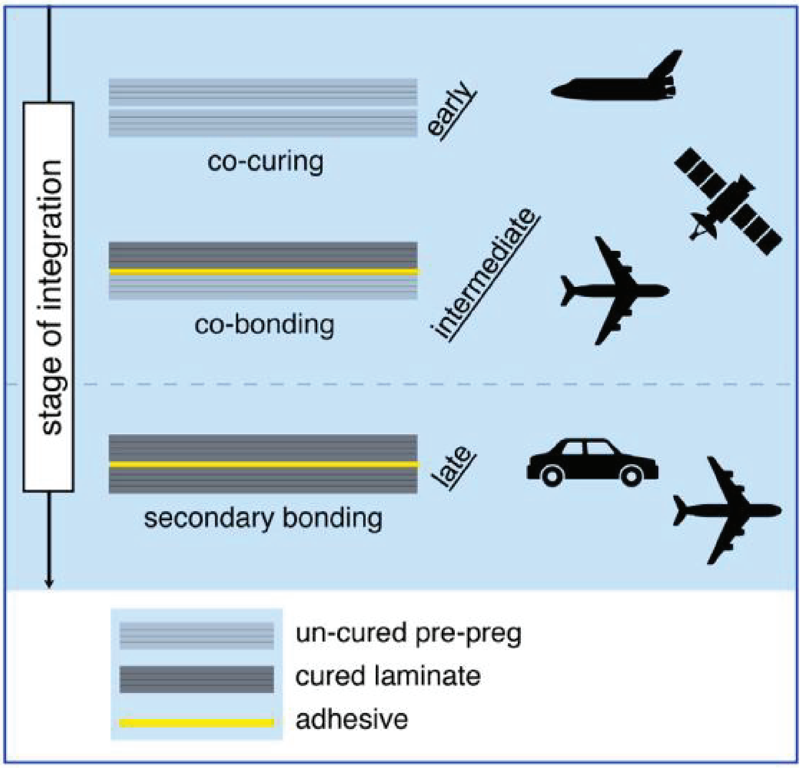

Figure 1.8. Joining techniques for bonded joint (Source: Yudhanto, A., et al., 2021)

In the beginning co-curing and co-bonding was using for manufacturing of aircrafts due to their well bonding properties than secondary bonding technique could have a chance to using because of disadvantage as needing expensive tools for applying co-curing and co-bonding techniques and due to advantages of it such as faster assembly, lower manufacturing cost, provide more flexibility on design, more versatile etc. In these days, all these techniques are widely used in aerospace and automotive industries. Apart from all these advantages, these bonding techniques rely on some factors which as good as adhesive types, adherend materials, joint geometry, environment, curing process and surface treatment which is the most critical one.[11]

In addition, six different failure modes characteristics exist for adhesive joints. These are adhesive failure, cohesive failure, thin-layer cohesive failure, fiber-tear failure, light-tear failure, and stock-break failure as illustrated from figure 1.9.[10] However, adhesive failure which occurs between the interface of the adhesive and one of the adherent and cohesive failure that results in physical separation from the adhesive layer on both adherent surfaces are two of the most common failure mechanisms among all failure mechanisms. Also, adhesive joint failures are generally not completely adhesive or cohesive but rather a mixture of the two. Nevertheless, ideal failure is determined as 100% cohesive.[12]

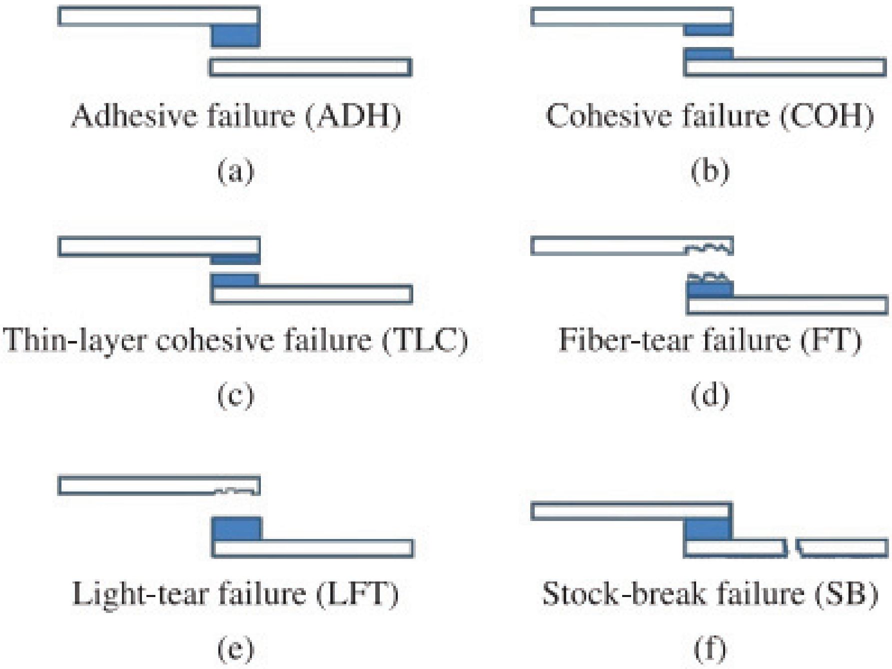
Adhesive failure (ADH)
(a)
Cohesive failure (COH)
(b)
Thin-layer cohesive failure (TLC)
(c)
Fiber-tear failure (FT)
(d)
Light-tear failure (LFT)
(e)
Stock-break failure (SB)
(f)

Figure 1.9. Figurations of failure modes characteristic of adhesive joints
(Source: Quini, J., et al., 2012)

1.5. Adhesion Surface Treatment Methods

In these days composite materials are used in almost all new technology fields due to their advantages such as light weight, lower manufacturing costs, flexibility of design etc. One of the most popular materials which are being used automotive, aerospace and defence industries are high-performance thermoset composites such as carbon fiber reinforced plastics also known as CFRP, and glass fiber reinforced plastics also known as GFRP. Especially in industries which priority of them is not cost, main purpose is reach products that have same strength with lower weight aerospace and defence generally CFRP is preferred.[13,14]

The characteristics of CFRP, including anisotropy, non-homogeneity, and the presence of hard-reinforced fibres, make adhesive joints an optimal solution for CFRP applications. Surface contamination and thicker resin layer than normal are critical points which have negatively effect on the quality of adhesive bonding strength.[14] Depending on this, surface treatment is one of the most critical factors which rely on reach better bonding strength.[11]

In general, surface treatments are examined in two main groups are called mechanical surface treatment and chemical surface treatment.[15] Peel ply, solvent cleaning, sanding, blasting, etching, plasma, chemical functionalization, and laser treatment are some of the most common surface treatments used to achieve a better quality of adhesive bonding for thermoset based composites. All these surface treatment methods have some disadvantages as like they have great advantages. Surface treatments are chosen depends on materials using in which application and in which industry moreover which properties of material are important for the application. Mechanical interlocking, adsorption, diffusion, and electrostatic adhesion are called the effects of the surface treatments which are given above on adhesion mechanisms. These adhesion mechanisms are shown in figure 1.10 above.[11]

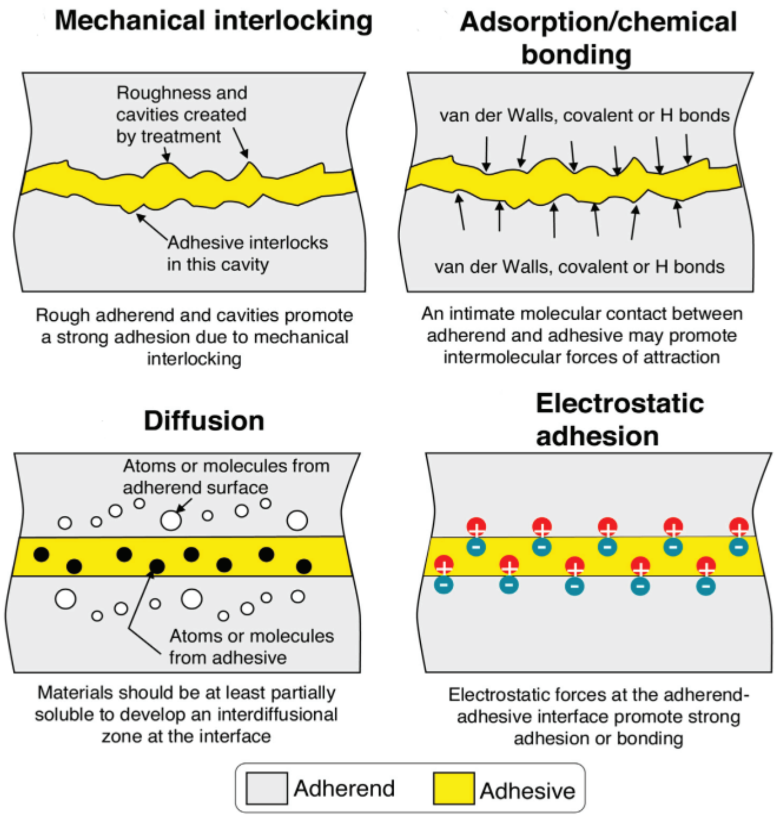

Figure 1.10. Adhesion mechanisms (Source: Yudhanto, A., et al., 2021)

1.5.1. Laser Surface Treatment

The use of laser radiation is perfectly fit for adhesive joint in CFRP plates. Selective resin removal without detrimental effects on fibers and at the same time removing contamination from the surface which are the biggest problems when using conventional joining methods and common pre-treatment methods such as peel ply, sand blasting, grinding and contaminated surface are basically the main reasons for using laser surface treatment.[8,14]

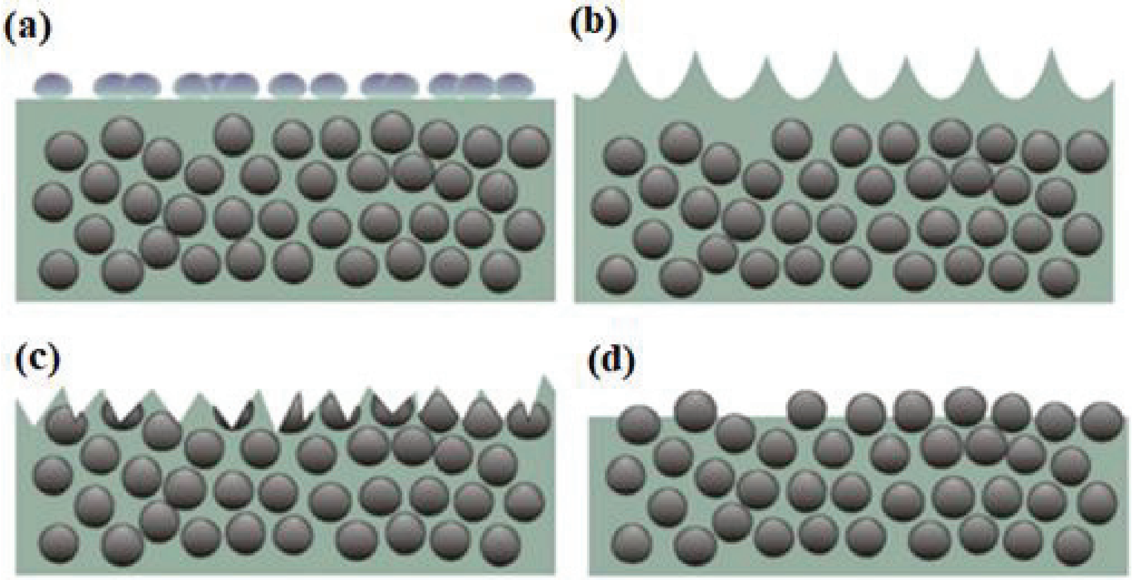

Figure 1.11. Different surface treatment and their effects on the adhesion surface; (a) contaminated surface, (b) peel ply, (c) grinding/sand blasting, (d) laser surface treatment (Source: Sun, C., et al., 2018)

Generally, increasing surface roughness and surface wettability are the similar aims to be achieved when applying surface treatment methods. These targets are reachable with using mechanical and chemical surface treatments, beside it some disadvantages become apparent with them such as for mechanical surface treatments are difficulty of process parameters control and for chemical surface treatments are inefficiency and environmental pollution. Laser surface treatment is becoming increasingly popular due to the elimination of several disadvantages and the provision of a number of important virtues. These include the increase in the bonding area, improvement in surface wettability, removal of contaminations from the surface and the avoidance of damage to carbon fibres when undertaking this process. As a result of applying laser surface treatment the strength of the adhesive joint increase. Different surface treatments and their effects on the adhesion surface are shown in figure 1.11.[15]

Different laser source types are used such as CO_2 laser, Ultra-violet (UV) laser and Infrared (IR) laser for surface treatment.[8,14] The CO_2 laser source is applicable fast on the other hands is occurred reducing the mechanical properties of the material because of inducing more heat into the material. The resin matrix and contaminations which on material surface are removed from the surface of the composite material without any damage to fibers with using UV laser.[16,17] Nevertheless, laser surface treatment by using UV has limited adoption to automation process and the main reason for this is that UV laser cannot be guided by optical fibers. Nonetheless, optical fibers can guide to IR lasers and IR lasers are perfect for automation.[17]

1.5.2. Electrospinning Method

Electrospinning method is not a new technique for the producing nanofibers. For the first time, this technique which is using for produce nanofibers from solution was reported for a patent issued in 1934 by A.Formhals.[18]

There are basically four main components for using this technique. These are a collector which can be different geometries depends on the project that using on, a capillary tube which contains the solution that using in process, a high voltage supplier which creates an electric field on the capillary tube and the collector and lastly a syringe pump which feeds the solution to the system. Schematic design of electrospinning technique is given in figure 1.12.[19]

The process of the electrospinning technique starts with preparing of the polymer solution. Different types of polymers and solvents can be useable in electrospinning technique. For preparing polymer solution step has some critical point exactly in here. There are concentrations of polymer solution and which types of solvent must be used together with which types of polymers. This information can be found in the literature. The polymer solution is infilled in capillary tube after it is prepared and the electric field which is created by high voltage supplier is subjected to the end of the capillary tube. The electric field creates induction on the surface of the polymer solution which held under surface tension. With increasing of intensity of the electric field shape of the polymer solution which at the tip of the capillary tube change from hemispherical to conical which as known as Taylor cone. When intensity of electric field reaches critical value which electrostatic force overcome to surface tension, a polymer jet, is charged, is ejected from the needle of the capillary tube to collector which is charged with oppositely by the high voltage supplier. Meanwhile, the solvent, inside of polymer solution, evaporates when it travels which from the needle of the capillary tube to the collector in the air and leave behind just a charged polymer fiber. As a result, the discharged polymer jet solidifies.[20]

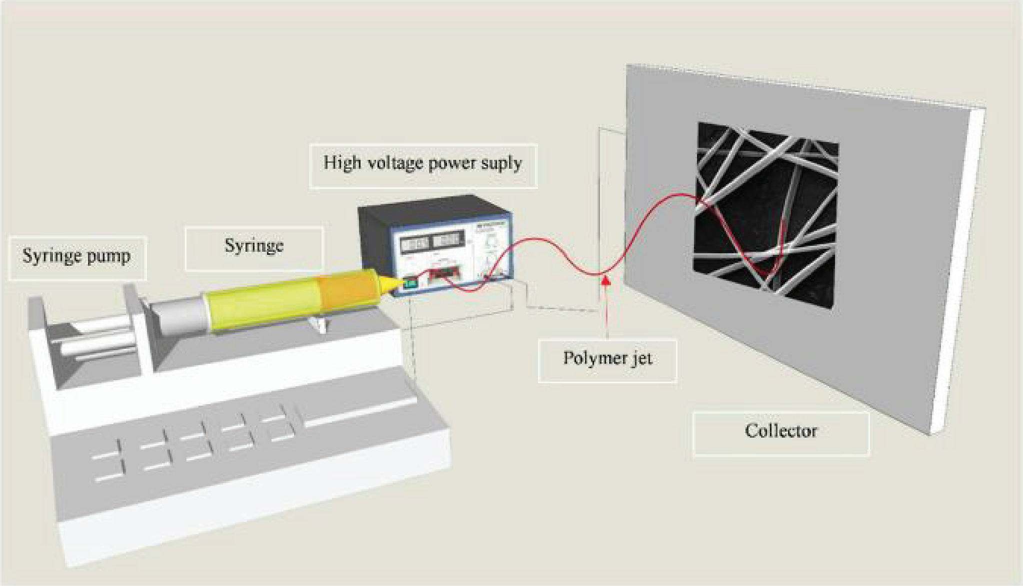

Figure 1.12. Schematic design of electrospinning technique
(Source: Rostamabadi, et al., 2020)

In addition, some parameters which are listed in three main variables effect on electrospinning process. These are electrospinning conditions such as humidity, air flow, temperature and ambient parameters, process parameters as like the distance between the needle of the capillary tube and the collector, the intensity of the applied electric field and the applied flow rate, solution attributes such as viscosity, concentration, conductivity, homogeneity, and surface tension.[19]

1.6. Fatigue

Fatigue which is a failure form name comes from the material that is subjected to stresses that are repeatedly applied a large number of times becomes tired from. Most of structure which use in different industries such as aerospace, defence etc. work under subjecting dynamic and fluctuating stresses on them. Under these conditions arise possibilities about failure which is called fatigue failure occurring dramatically lower than the tensile or even yield strength in some cases.

Fatigue is a significant failure form because of it is catastrophic and sneaky. It is a brittle failure even for ductile material and it occurs suddenly. In addition, the applied stress and the surface which has fracture are usually perpendicular to each other in a fatigue failure.[2,21]

1.6.1. Fatigue Variables

In nature, structures may be subjected to various types of stress, including axial, flexural, and torsional stress. Generally, there are three possibilities available for fluctuating stress-time modes: these are a reversed stress cycle which has sinusoidal graph between the maximum and the minimum stress and the mean stress is zero as illustrated in figure 1.13(a), a repeated stress cycle which has sinusoidal graph between the maximum and the minimum stress and the mean stress is different value from zero as illustrated in figure 1.13(b), and lastly a random stress cycle as it is understood from the name of it applied stress completely at random as also illustrated in figure 1.13(c). Furthermore, equations are given below to calculation of some variables such as mean stress,

$$\sigma_m = \frac{\sigma_{max}+\sigma_{min}}{2} \tag{1.1}$$

range of stress,

$$\sigma_r = \sigma_{max} - \sigma_{min} \tag{1.2}$$

stress amplitude,

$$\sigma_a = \frac{\sigma_r}{2} = \frac{\sigma_{max}-\sigma_{min}}{2} \tag{1.3}$$

and finally stress ratio

$$R = \frac{\sigma_{min}}{\sigma_{max}} \tag{1.4}$$

which are used in characterizing fluctuating stress-time graphs.[2]

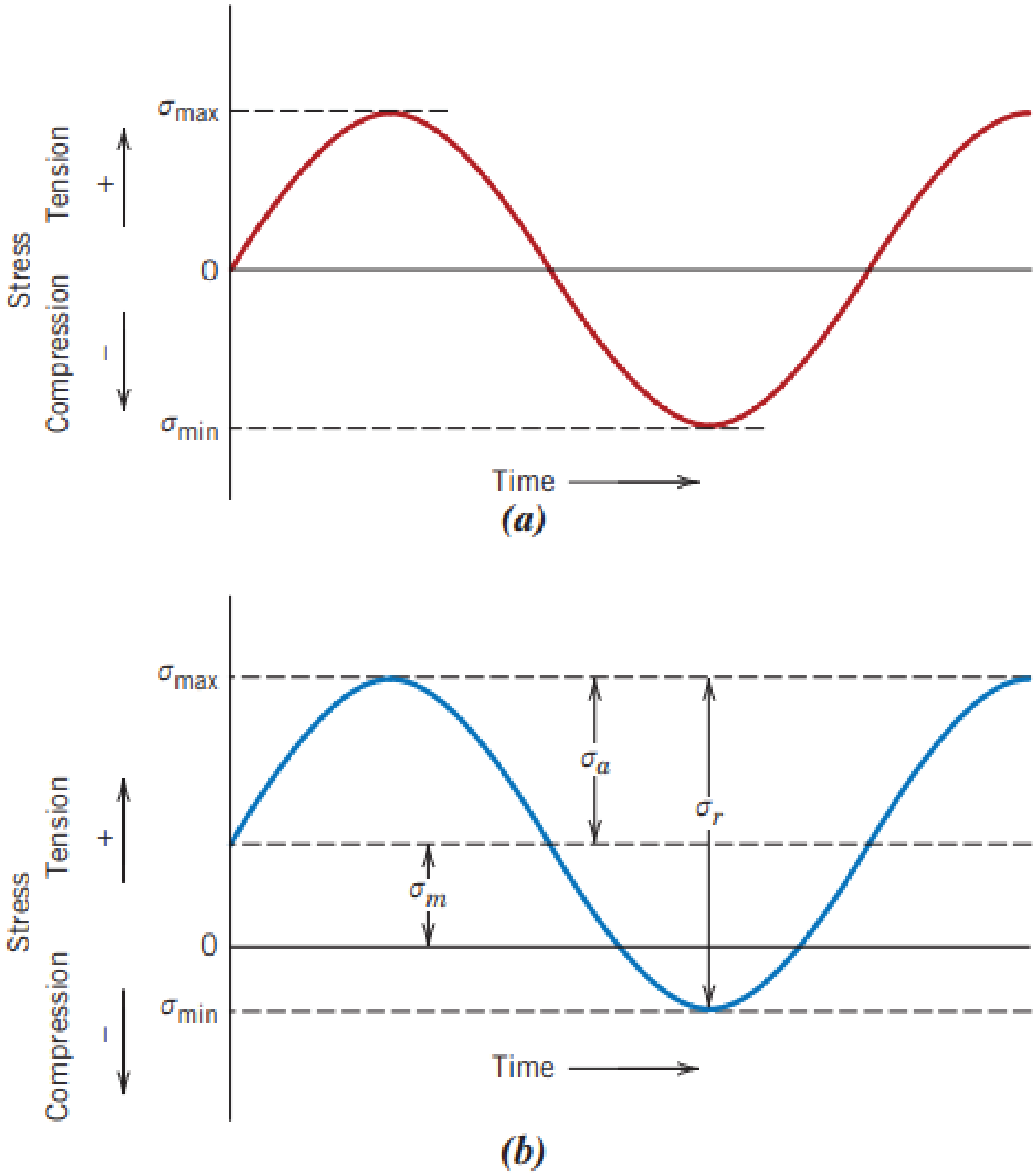

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Figure 1.13. Different fluctuating stress-time modes; (a) reversed stress cycle, (b) repeated stress cycle, (c) random stress cycle (Source: Callister, 2018)

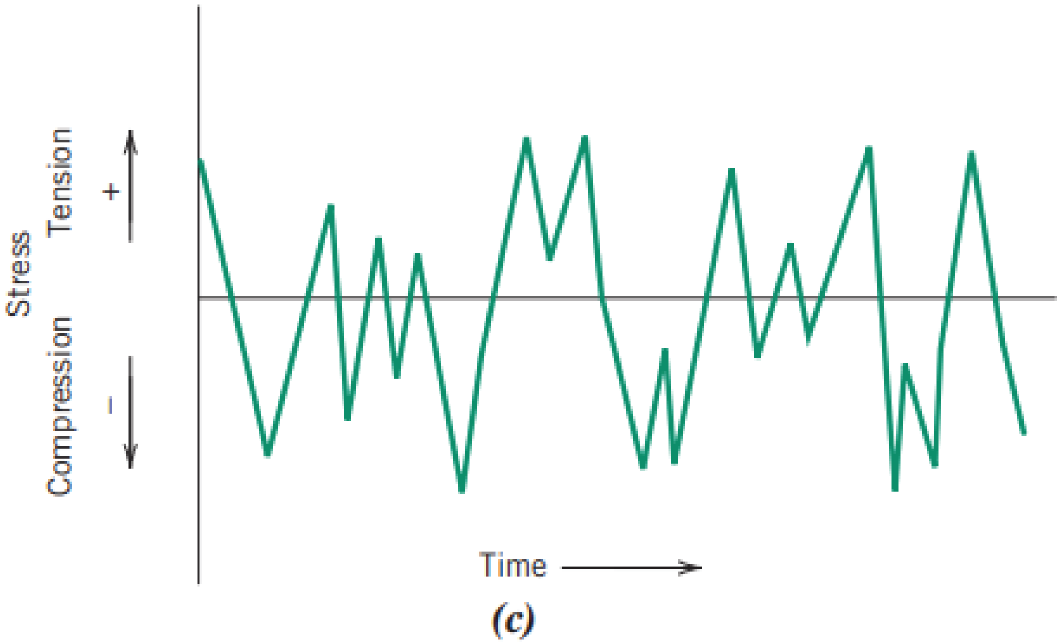


Figure 1.13. (cont.)

1.6.2. Wöhler Curve

Albert Wöhler investigated fatigue failure which some of the first notable one on railroad axles and articulated basic principle of fatigue failures in the middle of the 19th century.[21] The name of Wöhler curve comes from Albert Wöhler because of these studying on fatigue. Wöhler curve also known as S-N curve due to consist of cycling stress which is represented by S and fatigue life which is represented by N. Representative S-N curves are shown in figure 1.14.[22]

As it seen from figure 1.14(a), plotting of the S-N curve go toward to horizontal after reach a dotted line which known as the fatigue or endurance limit. The endurance limit represents a critical stress value. Apply any stress under endurance limit on specimen which made from ferrous material, specimen do not have fatigue failure in infinite number of cycles. However, this limit is not valid for non-ferrous materials. It is observed that the S-N curve which are plotted for the non-ferrous materials, continuously descend with an increasing number of cycles as shown in figure 1.14(b). For these kinds of materials, fatigue strength which is the stress level which is occurred fatigue failure in specified number of cycles is the term to consider.[2,22]

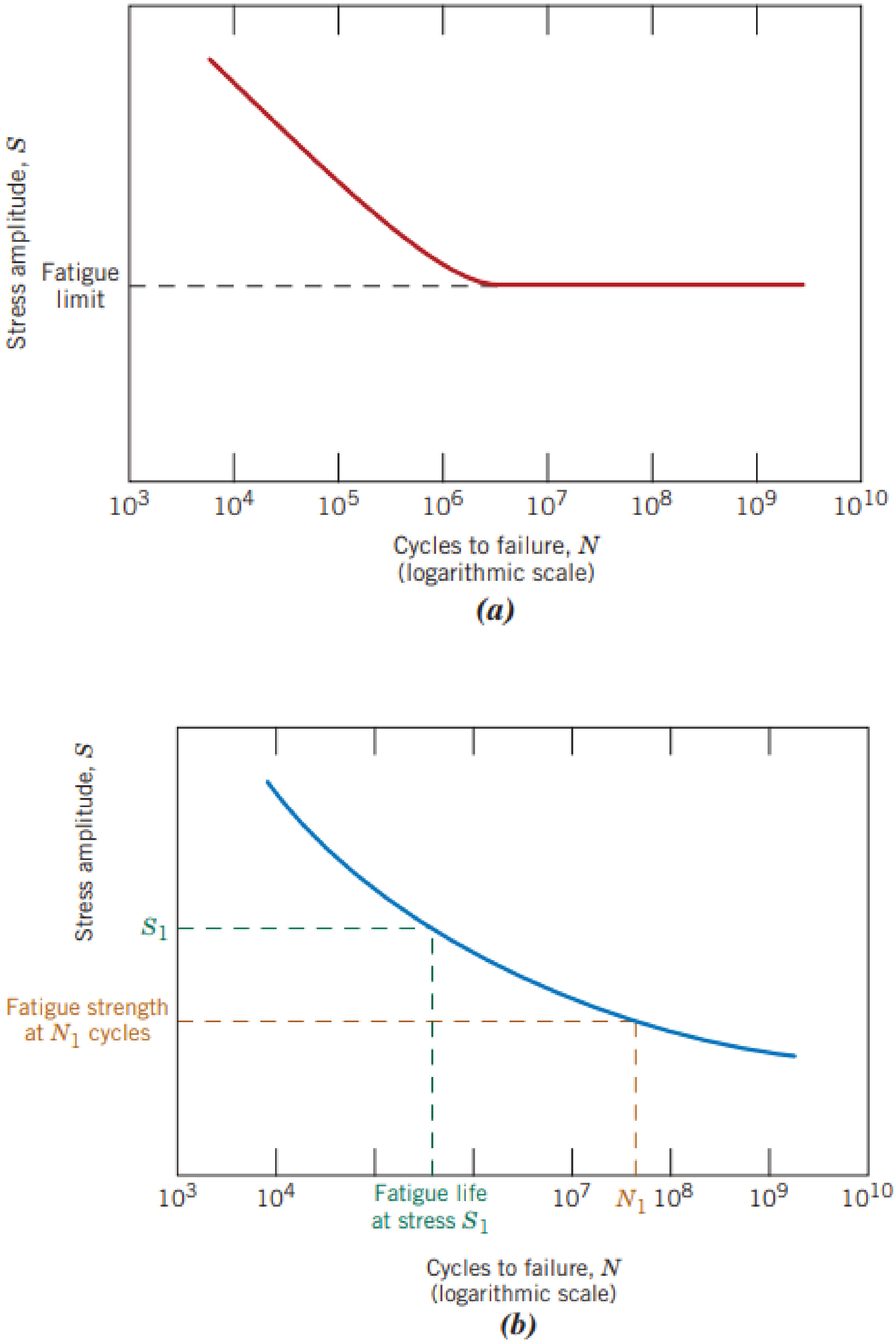


Figure 1.14. Representative S-N curv for (a) a material which displays endurance limit, (b) a material which does not display endurance limit (Source: Callister, 2018)

1.6.3. Fatigue of Composites

Fatigue failure in metals is noteworthy as it constitutes approximately 90% of all metal failures. The fatigue process in metallic materials has three distinct phases. There is crack initiation, crack propagation and final failure. As understood from these phases, fatigue failure in metallic materials starts with a small single crack initiation and progresses with crack growth until the critical size of the crack is reached and finally catastrophic failure occurs.[2,21,22] However as unlike metals and their alloys, composite materials are anisotropic and inhomogeneous. In composites, damage does not accumulate in a localised manner and therefore fatigue failure does not generally occur with the initiation and growth of a single crack. Instead, the process of accumulating damage involves microstructural mechanisms such as fibre breakage and matrix cracking, transverse-ply cracking, debonding and delamination. These phenomena can occur independently or interactively in different cases.[23]

Most composite materials suffer damage during early stages of cyclic loading. This damage is distributed along to the stressed region. Later in life during cycle, fatigue failure occurs when as an effect of great amount of damage accumulated in some region residual load bearing capacity of composite reduce to the stress which maximum loading in fatigue cycles. Moreover, the fatigue behaviour of reinforced plastics is influenced by several factors, such as fibre types, the matrix, environment, interleaving, loading conditions, and more.[23]

1.7. Objectives

In this study, the aim was investigated to the effect of improvements of the joint area performance on the structural fiber-reinforced composite parts on fatigue life in aerospace industry. These innovative improvements are applying electrospinning and laser surface treatment on the surface which use for adhesive joint. In the project, electrospinning and laser surface treatment applied on joint area than increasing in mechanical performance of composite structures are observed. In studies from the project, optimum polymer solution percentage and optimum laser offset values are found. In this study, fatigue tests realised with using lap shear test results which come from optimum parameters for both electrospinning and laser surface treatment. The results of fatigue test were compared with the results of specimens which do not apply any specific method on joint area as called reference for the observe the effect of innovative improvements on fatigue life.

CHAPTER 2

LITERATURE REVIEW

The use of composite materials in the aerospace and defence industries is widespread and expected to grow significantly over the next two decades. The primary reason for this is the superior strength-to-weight ratio of composite materials compared to metals. This property of composites also offers additional benefits such as lower fuel consumption, reduced emissions, and improved aerodynamic efficiency. Composite materials have become increasingly attractive due to their superior properties. These properties include tailorable mechanical properties, better corrosion resistance than metals, and excellent fatigue resistance. Additionally, the lightweight nature of composites provides higher maximum speeds and better manoeuvrability. These advantages make composites attractive not only in civil aviation but also in military aviation.[24,25]

The most common methods of joining composites to structures are mechanical fastening and adhesive bonding. Adhesive joints offer several advantages, including better stress distribution, durability, and lightweight properties. Additionally, they are undetectable by radar, which is important for military and defence industries. When using polymer-based composites with adhesive bonding, one of the most important points is applying surface treatment. This is due to the low wettability and surface energy of polymers. Several researchers have studied the application of surface modification techniques on composites, such as solvent cleaning, peel ply, abrasion, and laser treatment. The purpose of these studies was to remove contamination from the adhesion surface, increase roughness and wettability, and thereby improve bond strength.[26] Nattapat et al. 2015, applied laser surface treatment to the top resin layer of the CFRP. They uniformly and linearly removed the resin layer without exceeding the damage threshold of the fibers under the resin layer. In this study, the adhesive bond shear strength of a couple of specimens that underwent different surface modifications was compared. The specimens that underwent laser surface treatment achieved the highest shear bonding strength.[27] Çoban et al. 2019, in their studies showed that laser surface treatment is suitable to improve the bond strength of composites. The study also revealed that some parameters need to be optimised such as laser pattern, laser depth, number of laser shots and scribe pattern to achieve the best result.[28] In a study by Sun and colleagues, laser treatment was found to have a positive effect on the bonding strength of CFRP joints by improving the adhesion surface. The study identified the optimum laser ablation parameters and applied them to the bonding surfaces. As a result of the laser treatment, the resin layer above the carbon fibers was successfully removed without damaging the fibers, thereby increasing the bonding area. The lap shear strength of the adhesive increased as the bonding area between the adhesive and CFRP part increased.[15]

Polymer-based composites can fail due to various damage mechanisms depending on design parameters and loads. These mechanisms include matrix cracking, fiber breakage, and delamination. Delamination is defined the most catastrophic failure mechanism, as the composite loses its load-carrying ability after it occurs. Nanoparticle reinforcement is preferred to increase resistance to delamination. Electrospinning has been the preferred technique for producing nanofibers for the last two decades.[29] Beylergil et al. 2017, investigated the effect of using electrospun PA66 nanofibers as an interleaf material for CF/EP composites. The nanofibers were directly collected on carbon fabrics using the electrospinning process. The study found a 50% improvement in fracture toughness, a 15% improvement in compressive strength, and an 18% improvement in Charpy impact energy compared to reference specimens. However, the use of nanofibers resulted in a decrease in tensile strength.[30] Esenoğlu and colleagues conducted a study on electrospinning for CFRP parts. The study investigated the effects of different electrospinning solutions, adhesive thicknesses, and durations on the adhesion surface. To demonstrate the effect of electrospinning PA66 on the adhesion surface, single lap shear tests were performed, resulting in a 40% improvement compared to the reference specimens. The study found that using electrospun nanofibers on the adhesion surface not only increased bonding strength but also changed the type of adhesive failure from premature adhesive failure to cohesive failure. This resulted in an improvement in the adhesion performance of the composite.[31]

Materials used in engineering applications are subjected to both static and cyclic loadings. One of the most important tests for designing materials is the fatigue test, which examines the cyclic behaviour of materials. The behaviour of composite materials under fatigue loading differs significantly from that of metals. Under fatigue loading, metals experience crack initiation and propagation leading to fracture. However, in the case of composite materials, several micro cracks initiate, resulting in various types of fatigue damage. The fibre volume fraction is an important parameter for the fatigue strength of composites. Increasing the fibre volume of composites does not always have a positive effect. In some cases, if the fibre volume continues to increase after a certain level, the fatigue strength of the composites decreases due to insufficient resin to grip the fibres. Parameters such as fibre orientation, loading type, manufacturing process, frequency, mean stress, and environment can all affect the fatigue behaviour of fibre-reinforced polymer composites.[32] Two models are commonly used for fatigue damage in the literature: the residual strength approach and the residual stiffness approach. The residual stiffness approach divides the stiffness degradation of mostly fibre reinforced composite materials into three stages: initial decline, gradual reduction, and final failure, as shown in Figure 2.1. Schulte et al. studied the tension-tension fatigue (R=0.1) behaviour of carbon/epoxy specimens with a stacking sequence of $[0,90,0,90]_{2s}$. This study examined the fatigue performance of specimens in relation to stiffness degradation, as monitored by Schulte. The study identified three distinct regions. In the first region, a stiffness decreases of 2-5% was detected due to the development of transverse matrix cracking. In the second region, additional stiffness reduction of 1-5% was caused by the development of edge delamination and longitudinal cracks along the 0° fibres. In the final region, there was a sudden reduction in stiffness due to local damage progression.[33]

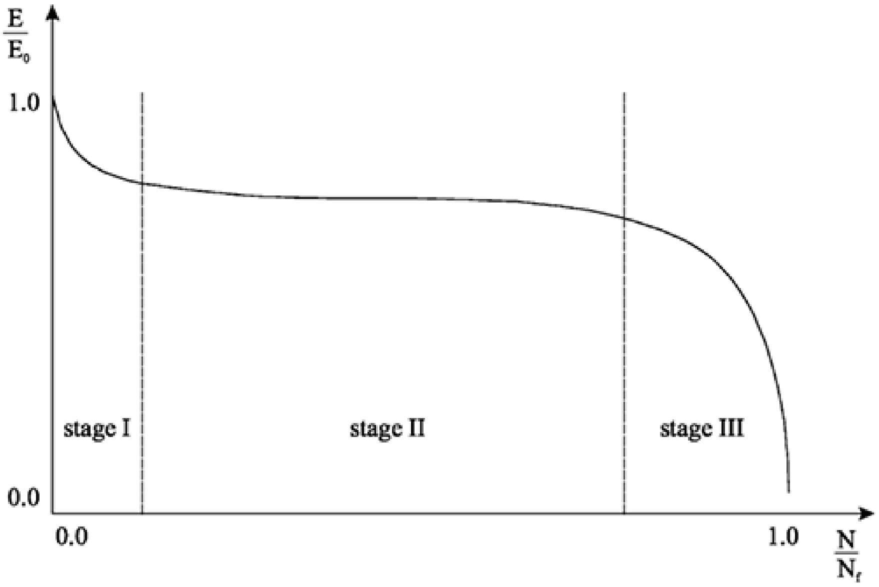


Figure 2.1. Usual stiffness degradation curve for fibre-reinforced composite materials (Source: Van Paepegem and Degrieck, et al., 2002)

Literature contains numerous studies on the fatigue behaviour of polymer matrix composites. However, only a few studies focus on the fatigue behaviour of carbon fibre reinforced polymer prepregs with adhesively bonded joints and the effect of surface applications on their fatigue behaviour. This is due to their increasing use in the aviation and defence industries.

In 2018, Pagano and his colleagues studied the fatigue performance of unidirectional laminates based on carbon-epoxy prepregs with a 57% fibre volume fraction. They used different stacking sequences, such as four, eight, and sixteen, to observe the effects on fatigue performance. Tension-tension fatigue tests were performed at a combination of 3 and 5 Hz frequencies and 0.1 and 0.5 stress ratios. Disintegration occurred in specimens around 25000 cycles when the stress ratio was 0.1. However, when the stress ratio was 0.5, specimens did not show any damage even after 100000 cycles. This study highlights the significant role that stress ratios between 0.1 and 0.5 play in the fatigue behaviour of unidirectional CFRP laminates.[34]

Quaresimin et al. studied the behaviour of adhesive single lap joint carbon-epoxy laminates under fatigue loading. They also investigated the effect of surface preparation, spew geometry, and overlap length on fatigue damage. The experiments were conducted under tension loading. The study found that adding a spew fillet at the end of the overlap resulted in more than a 25% improvement in fatigue strength. Additionally, increasing the overlap length on the adherends was found to increase fatigue strength. Fatigue damage evolution was analysed in this study. The analysis revealed the presence of a nucleation phase followed by crack propagation up to joint failure at the adhesive-adherend interphase.[35]

Numerous studies have investigated the effect of surface treatment on the bonding strength of CFRP. However, research on the influence of surface treatment on fatigue performance is scarce. In 2020, Park and his colleagues examined the effect of surface treatment on fatigue strength by using single lap bonded joints. CFRP plates were fabricated using unidirectional carbon fiber-epoxy prepreg materials with approximately 57.5% fiber volume fraction. The plates were made using the vacuum bag autoclave molding technique and a $[0°/45°/90°/-45°]_{3S}$ stacking sequence. To prepare the bonding surface of the single lap joint specimens, grit blasting, peel ply with grit blasting, and manual sanding were applied. The specimens' geometry was based on ASTM D5868 standard. The use of peel ply with grit blasting treatments resulted in higher surface roughness and energy. However, the fatigue strength did not improve as expected. This study shows that the expectation of improved strength performance as a benefit of surface treatment methods is deceptive.[36]

In recent years, laser surface treatment has been used to improve the bonding strength of CFRPs. Bello and his colleagues studied the effect of laser surface pre-treatment on fatigue crack growth of carbon fiber reinforced structures. They used unidirectional carbon-epoxy prepregs as the substrate and two-component epoxy for bonding the CFRP substrates. The laminates were manufactured with a $[0^\circ]_8$ stacking sequence. Fatigue tests were performed on specimens prepared based on the double cantilever beam test, with a stress ratio of 0.1 and a frequency of 10 Hz. The study involved treating CFRP substrates with a teflon film as reference specimens. Subsequently, the substrates were treated with a CO_2 laser in two different groups: low energy irradiation and high energy irradiation. Low energy irradiation resulted in a clean surface with partially exposed fibers. High energy irradiation of CFRP resulted in the complete removal of epoxy over the fibres, leaving them exposed. Test results showed that both laser treatment groups achieved higher surface energy than the teflon treatment. Additionally, the crack growth rate sensitivity of the high energy irradiation treatment was found to be higher than that of the reference specimens.[37]

Various methods have been proposed in the literature to enhance the fatigue performance of bonding strength. Polat et al. investigated the modification of the matrix with nanoparticles to prevent sudden fracture in adhesive joints of composite structures under fatigue loading. They manufactured a carbon fiber composite laminate with a [0/90/0/90] stacking sequence using vacuum-assisted resin transfer molding technique. Graphene nanoplate-doped nylon 66 was used as the preferred nanoparticle and produced using the electrospinning method in three different groups: 1wt%, 3wt%, and 5wt% graphene nanoplate-doped nylon 66 nanofibers. Fatigue tests were performed with a single-axis periodic loading at a frequency of 10Hz and a stress ratio of 0, using five different loading levels: 20%, 30%, 40%, 50%, and 60% of the maximum shear strength of the specimens. Increasing fatigue life was determined as a result of tests conducted for each loading level and each weight percentage of nanoparticles.[38]

Brugo and his colleagues investigated the effect of adhesion surface nanofiber modification on delamination propagation in carbon fiber-epoxy resin composite laminates. Nylon 66 nanofibers were produced using the electrospinning method. Fatigue tests were performed using double cantilever beam specimens. The study found a 130% increase in delamination toughness and a 96% increase in crack growth under fatigue loading. In SEM images, the nanofibers were visible as they interleave on the adhesion surface, holding the matrix together.[39]

CHAPTER 3

EXPERIMENTAL

3.1. Materials

In this study, carbon fibre reinforced polymer (CFRP) composite laminates were produced using unidirectional (UD) prepregs with a carbon epoxy matrix, identified by the code M91/34/UD194/IM7-12K. The prepregs, which unit weight is 294 g/m^2, were used as the reinforcing material. Furthermore, a film adhesive known as FM300K was employed. Both prepregs and film adhesives were supplied by Turkish Aerospace Industries Incorporated (TAI). The raw materials used for preparing the electrospinning solution were Sigma Aldrich-429171 as polyamide 66 pellets, Sigma Aldrich-27001 as formic acid, and Sigma Aldrich-24216 as chloroform.

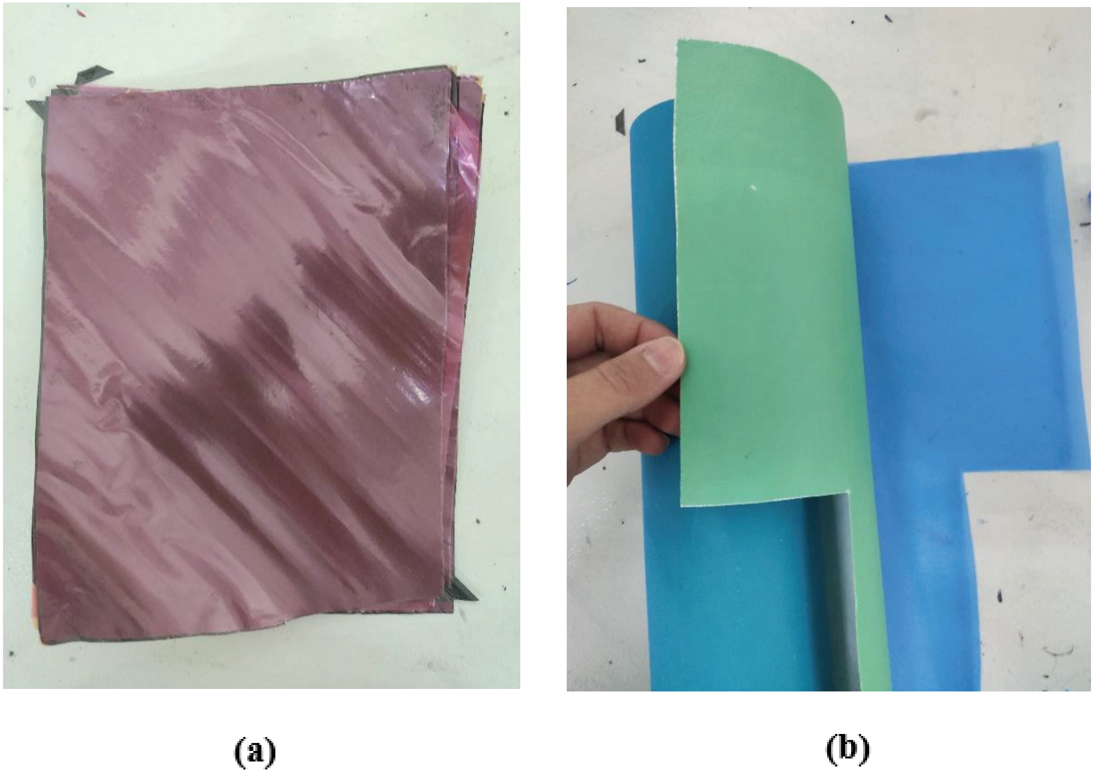

(a) (b)

Figure 3.1. Materials used for production of CFRP plates (a) M91/34/UD194/IM7-12K UD prepreg, (b) FM300K film adhesive

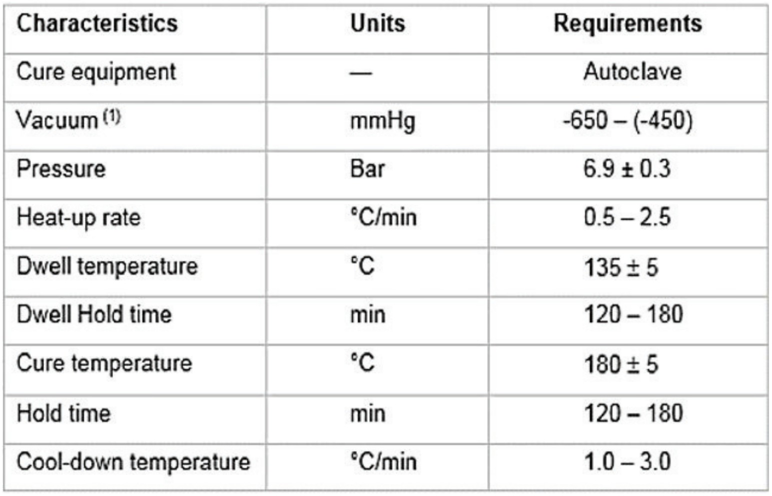
3.2. Manufacturing of Composite Plates

The composite laminates were produced using a $[45/-45/45/0/-45/90]_s$ stacking sequence of 12-layer prepregs each and fabricated using the autoclave technique. The CFRP composite laminates were fabricated in an autoclave provided by Turkish Aerospace Industries Incorporated. The process was carried out at a temperature of 180 ± 5°C and a pressure of 6.9 ± 0.3 bar for a duration of 120-180 minutes. Figure 3.2 shows the parameters used in the fabrication process.

The CFRP composite laminates were fabricated with final dimensions of 300 mm x 300 mm x 3 mm. Two distinct types of composite laminates were produced using the autoclave technique. The difference between them arises from the production of nanofibers on the bonding interface layer of laminates via electrospinning techniques on the surface. In addition, the composite laminates in the other group were manufactured without any specific processes applied. These laminates were used for reference and laser surface treatment groups. Laser surface treatments were applied on the bonding interface of the composite laminates that had already been manufactured. Table 3.1 provides some details on the materials used to fabricate the composite plates.

Some parameters are crucial in the applications of both electrospinning and laser treatment. One of these critical parameters is the weight percentage of polyamide 66 concentration in the solution for electrospinning, while for laser treatment, it is the laser offset distance.

(a)



Characteristics	Units	Requirements
Cure equipment	—	Autoclave
Vacuum (1)	mmHg	-650 – (-450)
Pressure	Bar	6.9 ± 0.3
Heat-up rate	°C/min	0.5 – 2.5
Dwell temperature	°C	135 ± 5
Dwell Hold time	min	120 – 180
Cure temperature	°C	180 ± 5
Hold time	min	120 – 180
Cool-down temperature	°C/min	1.0 – 3.0

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Figure 3.2. Selected parameters for producing CFRP composite laminates via autoclave are presented in (a) table, (b) graph

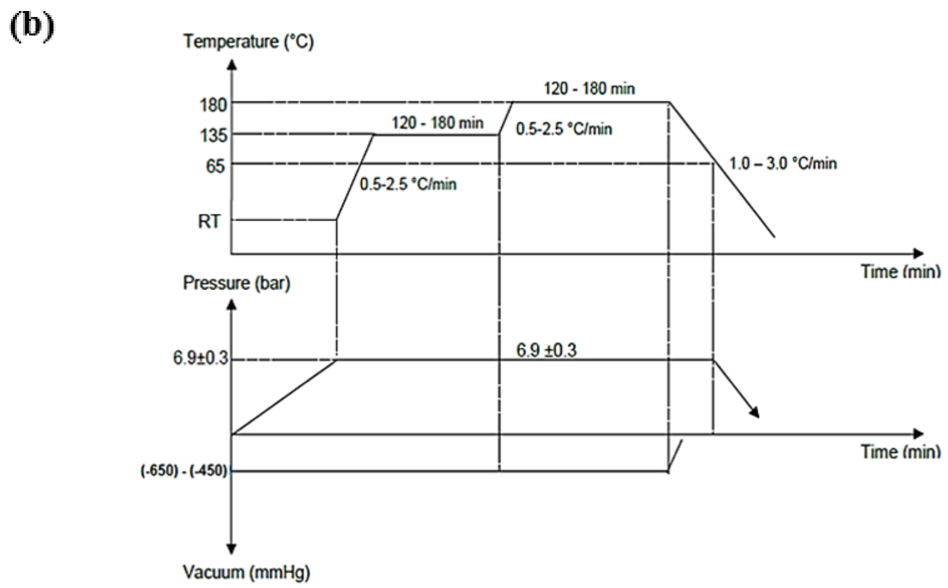


Figure 3.2. (cont.)

Table 3.1. Details of adherend materials

Applying Process	Material Name	Stacking Sequence	Ply
None	M91/34/UD194/IM7-12K	$[45/-45/45/0/-45/90]_s$	12
Electrospinning	M91/34/UD194/IM7-12K	$[45/-45/45/0/-45/90]_s$	12
Laser Treatment	M91/34/UD194/IM7-12K	$[45/-45/45/0/-45/90]_s$	12

The effect of different weight percentages of polyamide 66 concentration was investigated (10%, 12%, 14%, and 16%) on the production of nanofibers. The SEM images were examined, and it was concluded that the most homogeneous fibers were produced with 10% weight of PA66 in the solution.[40] The laser surface treatment parameters, namely power, speed, and frequency, were set to 20 W, 10000 mm/s, and 100 kHz, respectively. Additionally, laser offset was explored, a crucial parameter for removing epoxy from the surface and achieving optimal adhesion performance, at 0.15, 0.20 and 0.25mm.[41] The Izmir Institute of Technology's mechanical engineering laboratory utilised the INOVENSO PE300 device to produce nanofibers via electrospinning. Additionally, the FLAST-NanoMARK-50w IR-Yb (Ytterbium) fiber nanosecond laser device was employed for laser treatment. Both devices are illustrated in Figure 3.3 and 3.4.

Figure 3.3. INOVENSO PE300 device

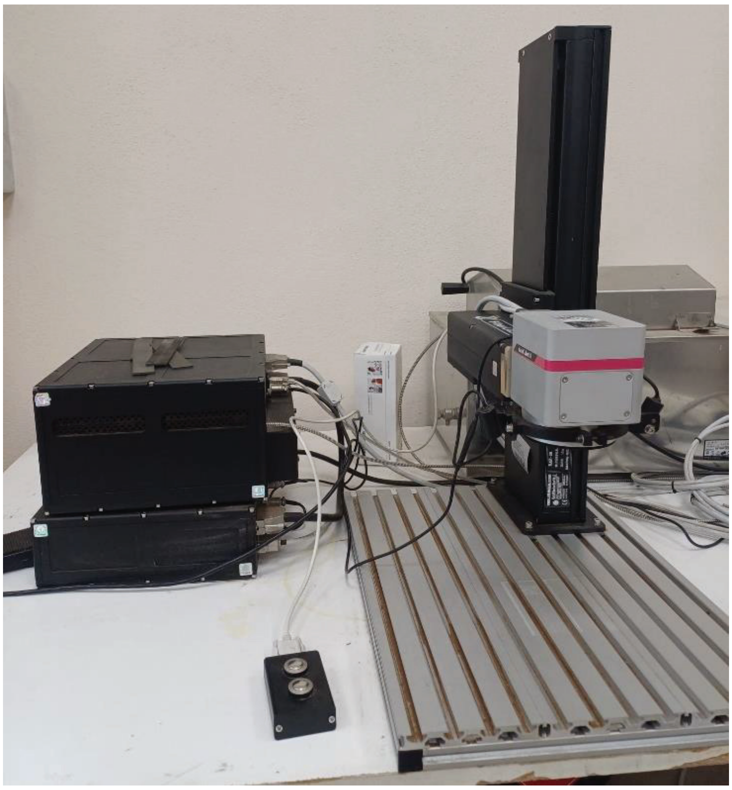


Figure 3.4. FLAST-NanoMARK-50w device

Figure 3.5 illustrates the production of CFRP composite plates using FM300K film adhesive and CFRP composite laminates that undergo special processes on adhesion surfaces, including electrospinning and laser treatment.

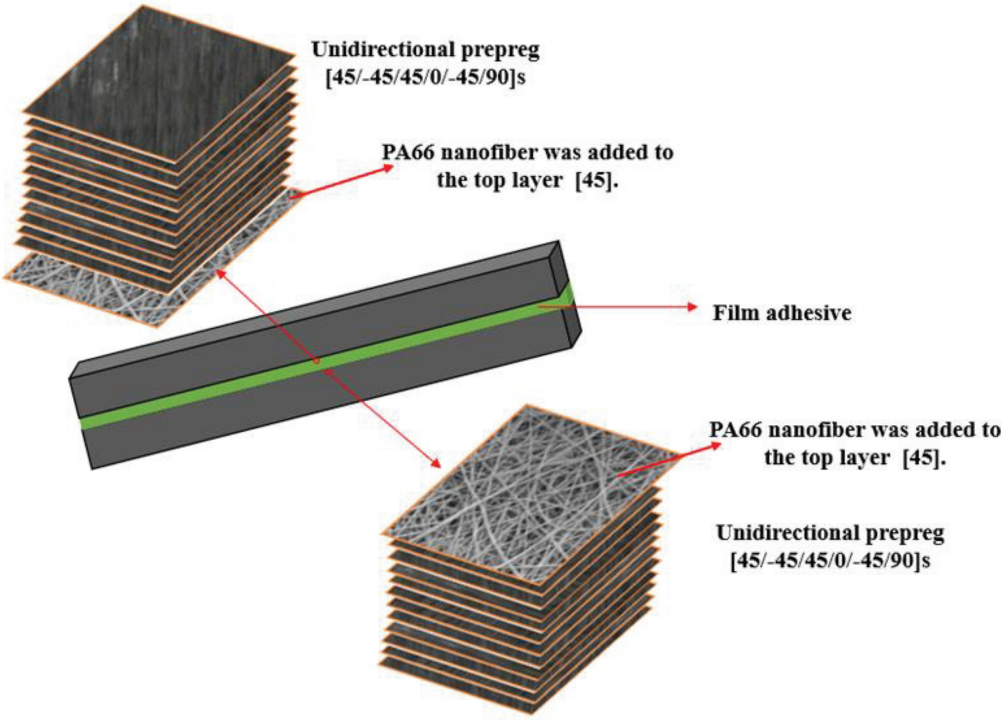
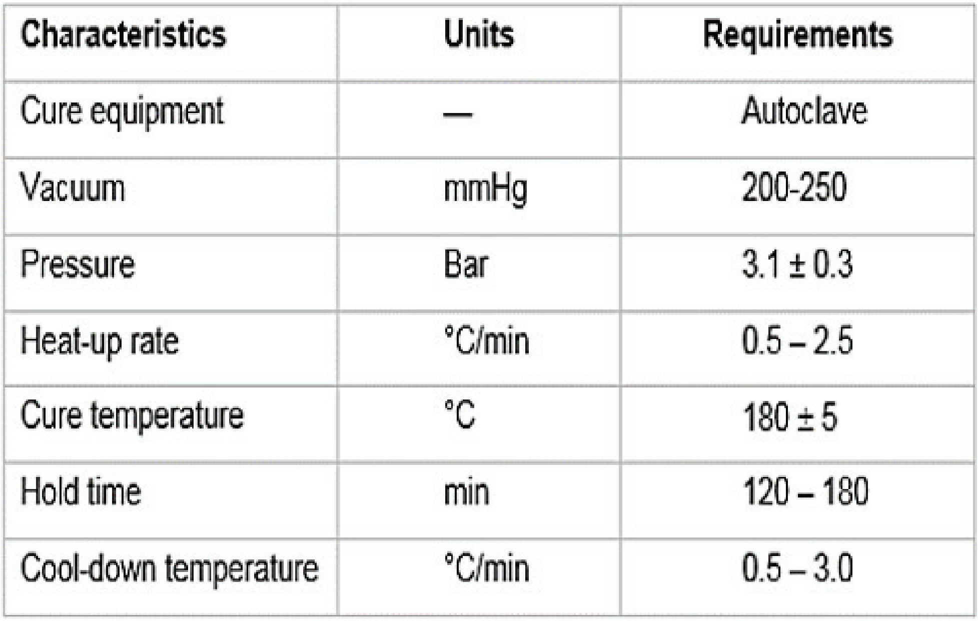


Figure 3.5. Schematic illustration of CFRP composite plate manufacture applied electrospinning

CFRP composite laminates, as shown in Table 3.1, were bonded using FM300K film adhesives and CFRP composite plates were produced as a result of the process. The adhesive films, selected at 3 ply and 0.60 mm, were placed between the composite laminates. Figure 3.6 illustrates the parameters, including curing temperature, curing time, and applied pressure, that are essential for the production of CFRP composite plates using the autoclave technique.

CFRP composite plates were cut to specified dimensions using a water-cooled diamond saw blade. The final specimens were then sanded and placed in a drying oven. The specimens were dried at 50°C for one hour. Finally, the specimens were machined to their final dimensions in accordance with ASTM standards using a CNC router. Figure 3.7 show the equipment used in the mechanical engineering laboratory at Izmir Institute of Technology, including the water-cooled diamond saw blade, the CNC router, and the drying oven.

(a)



Characteristics	Units	Requirements
Cure equipment	—	Autoclave
Vacuum	mmHg	200-250
Pressure	Bar	3.1 ± 0.3
Heat-up rate	°C/min	0.5 – 2.5
Cure temperature	°C	180 ± 5
Hold time	min	120 – 180
Cool-down temperature	°C/min	0.5 – 3.0

(b)

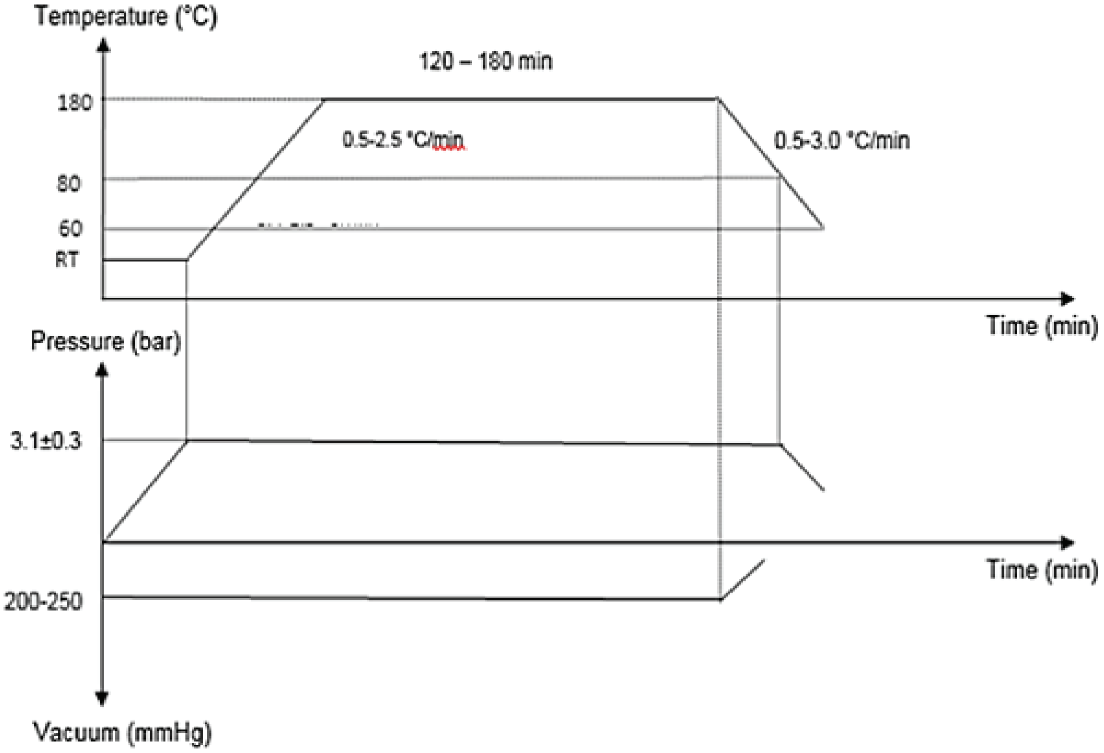


Figure 3.6. Selected parameters for joining CFRP composite laminates with FM300K in the autoclave technique are presented in (a) table, (b) graph

(cont. on next page)

Figure 3.7. The devices belonging to the laboratory of the Izmir Institute of Technology; (a) water-cooled diamond saw blade, (b) drying oven, (c) CNC router device

(c)

Figure 3.7. (cont.)

3.3. Mechanical Testing

Single lap shear tests were conducted on adhesively bonded CFRP composites to examine the effect of electrospinning and laser treatment applications on the mechanical performance of the adhesive bond between CFRP laminates. The mechanical tests were realized under atmospheric conditions and in accordance with the ASTM standards.

3.3.1. Single Lap Shear Tests

Single lap shear tests were conducted in accordance with ASTM D5868-01 standards using the MTS Landmark^{TM} Servohydraulic Test System. The loads were applied at a rate of 13 mm/min, as specified by the standard.[42]

Single lap shear tests were used to determine and compare bonding properties of reference, electrospinning, and laser treatment CFRP-CFRP composite specimens. Figure 3.8 illustrates the test specimens used for the single lap shear tests.[31]

3.4. Fatigue Testing

Fatigue tests were performed to examine the effect of adhesion surface treatments, such as electrospinning and laser treatment, on the fatigue performance of adhesively bonded carbon fiber-based polymer composites. The test samples were prepared with the same dimensions as the single lap shear tests.

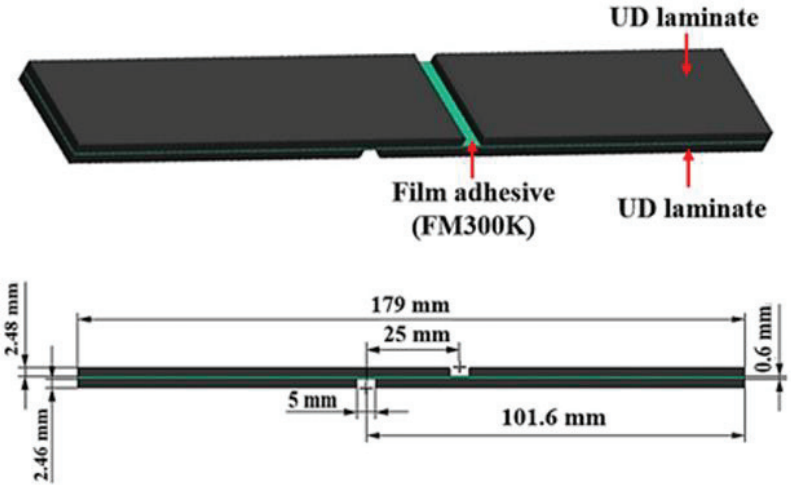


Figure 3.8. Single lap shear test specimens (Source: Esenoğlu, G., et al., 2022)

The fatigue tests were conducted in load control mode, with a fixed stress ratio (R) of 0.1 and a selected testing frequency of 2 Hz. The tests were completed at three different stress levels, which were 30%, 40%, and 50% of the average single lap shear strength. Fatigue tests were conducted on four groups of specimens with different surface pre-treatment methods, namely laser treatment, reference, electrospinning, and a second group of laser treated specimens. The effect of two different laser offset distances on fatigue performance was investigated. Figure 3.9 shows the fatigue test setup on the MTS Landmark^{TM} Servohydraulic Test System.

Figure 3.9. Fatigue test setup on the MTS Landmark^{TM} Servohydraulic Test System

CHAPTER 4

RESULTS AND DISCUSSION

In this chapter discusses the effect of surface treatment on bonding strength using the single lap shear test results of three different groups: reference, laser treated, and electrospinning. In addition, at the end of the chapter, the effect of the same surface treatment on fatigue life is discussed with SEM images, stiffness degradation and S-N curves.

4.1. Single Lap Shear Tests

The shear properties of composite specimens with applied surface treatment on the adhesion surface were investigated. Single lap shear test specimens were prepared according to ASTM D5868. To investigate the effect of surface treatment on the adhesion strength of composite specimens, manufactured with using FM300K film adhesive between CFRP composite laminates, were prepared with three different surface treatment groups: reference, laser surface treatment, and electrospinning.

The laser treatment was carried out using a FLAST-NanoMARK-50w, which is an IR-Yb (Ytterbium) fiber nanosecond laser. Two different laser offset parameters were used to remove epoxy from the surface, namely 0.15mm and 0.20mm, and they were sequentially named L1 and L2. Other parameters, other than the laser offset parameter, were fixed for all tests, and are shown in Table 4.1 below.

The specimens were produced by electrospinning PA66 onto the joining surface and curing it in an autoclave according to ASTM D5868. Nanofibers were produced using a 10% weight rate of PA66 in solution and coated onto the joining surface for 10 minutes with using the INOVENSO PE300. Coated prepregs were cured in an autoclave to produce CFRP composite laminates. Three layers of FM300K film adhesive were added between the composite laminates for manufacture CFRP-CFRP composite plates. Composite plates were produced at TAI using optimum parameters determined through laboratory tests at IZTECH.

The results of the single lap shear test for each group of specimens are given in force-displacement curves. Tables and graphs show the maximum single lap shear results for each specimen along with their standard deviations for illustrative purposes.

Table 4.1. Laser surface treatment parameters

Specimens / **Parameters**	**L1**	**L2**
Laser Offset Distance (mm)	0.15	0.20
Wavelength (nm)	1064	1064
Frequency (kHz)	100	100
Spot Diameter (nm)	30	30
Pulse Width (ns)	100	100
Scanning Speed (mm/s)	10000	10000
Power (W)	20	20

Specimens, which were already performed single lap shear, are illustrated in Figure 4.1, 4.2, 4.3 and 4.4. In addition, load-displacement graphs of them are given in Figure 4.5, 4.6, 4.7 and 4.8.

Figure 4.1. Single lap shear reference specimens

Figure 4.2. Single lap shear L1 specimens

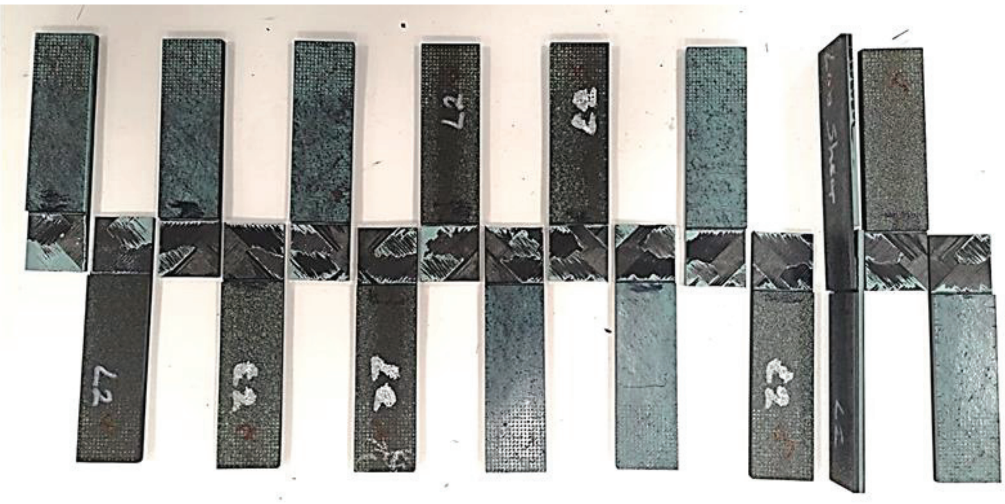


Figure 4.3. Single lap shear L2 specimens

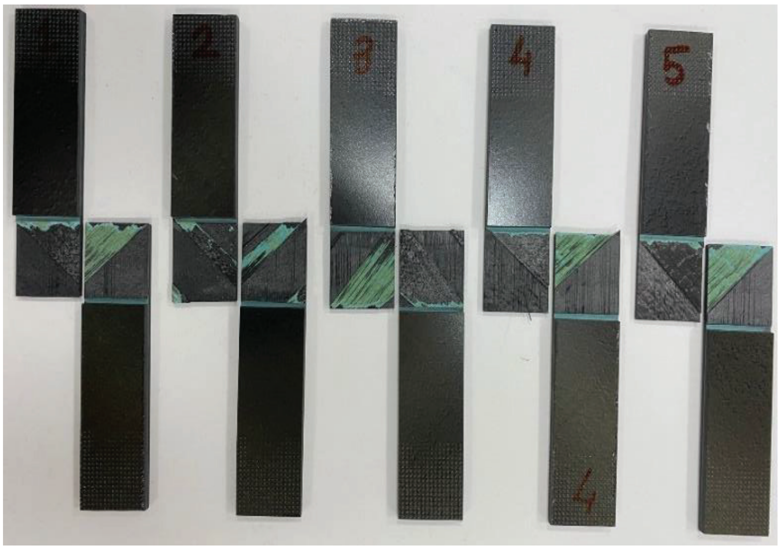


Figure 4.4. Single lap shear electrospinning specimens

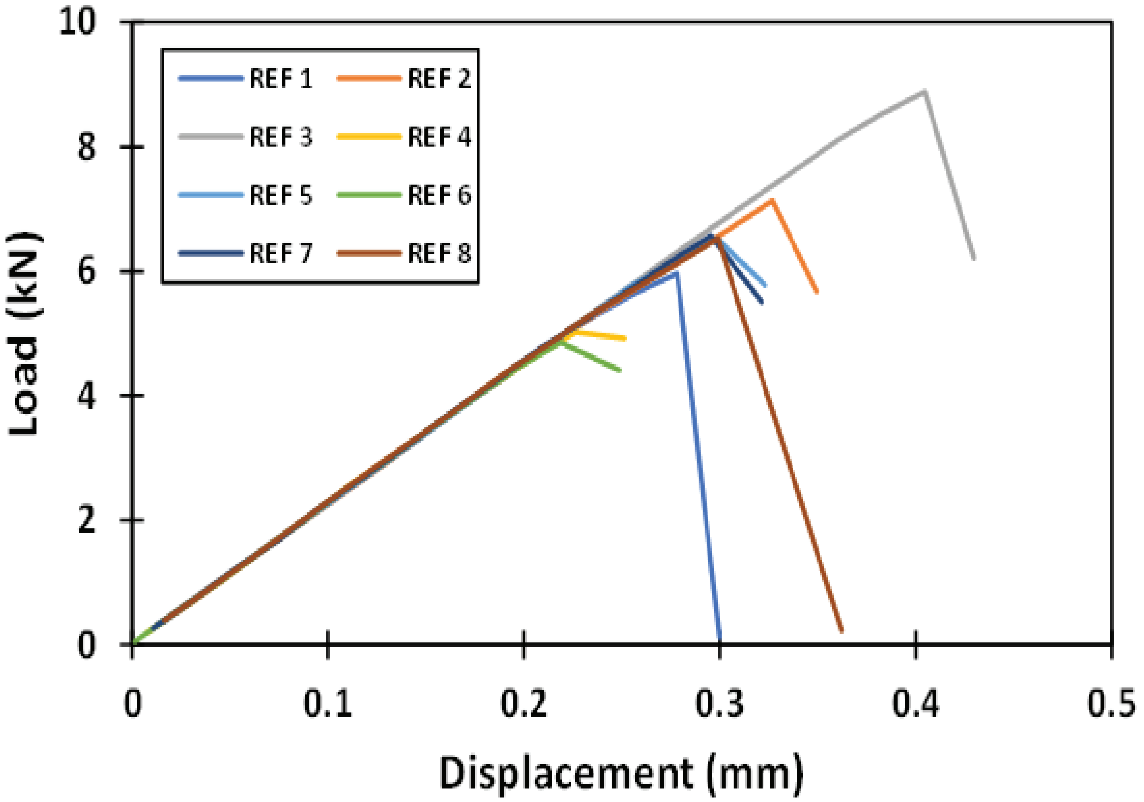


Figure 4.5. Load-displacement graph for reference specimens

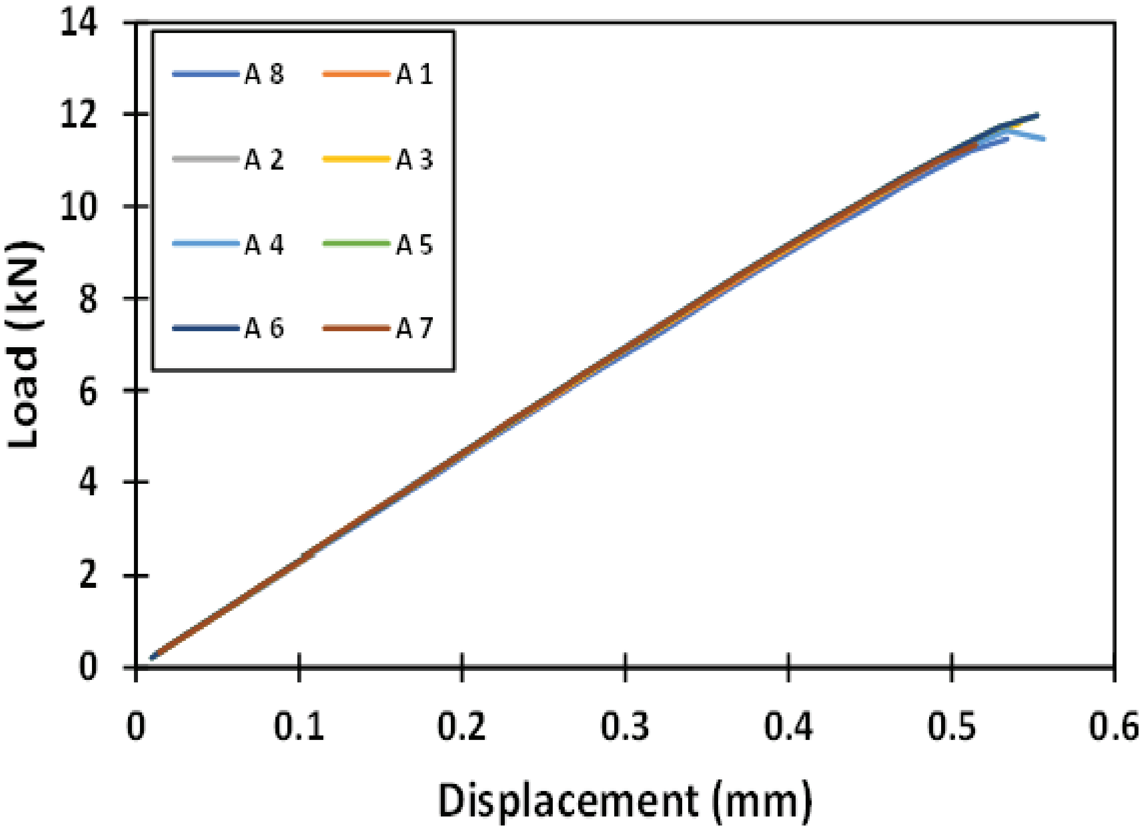


Figure 4.6. Load-displacement graph for L1 specimens

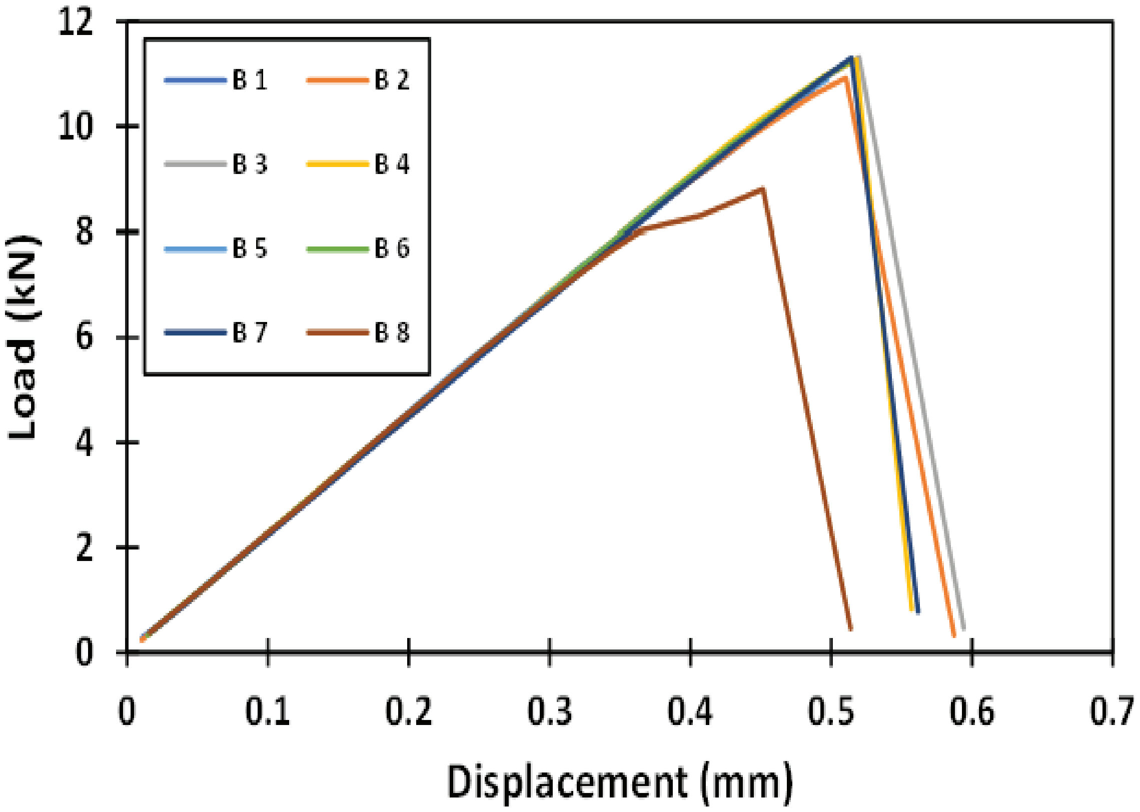


Figure 4.7. Load-displacement graph for L2 specimens

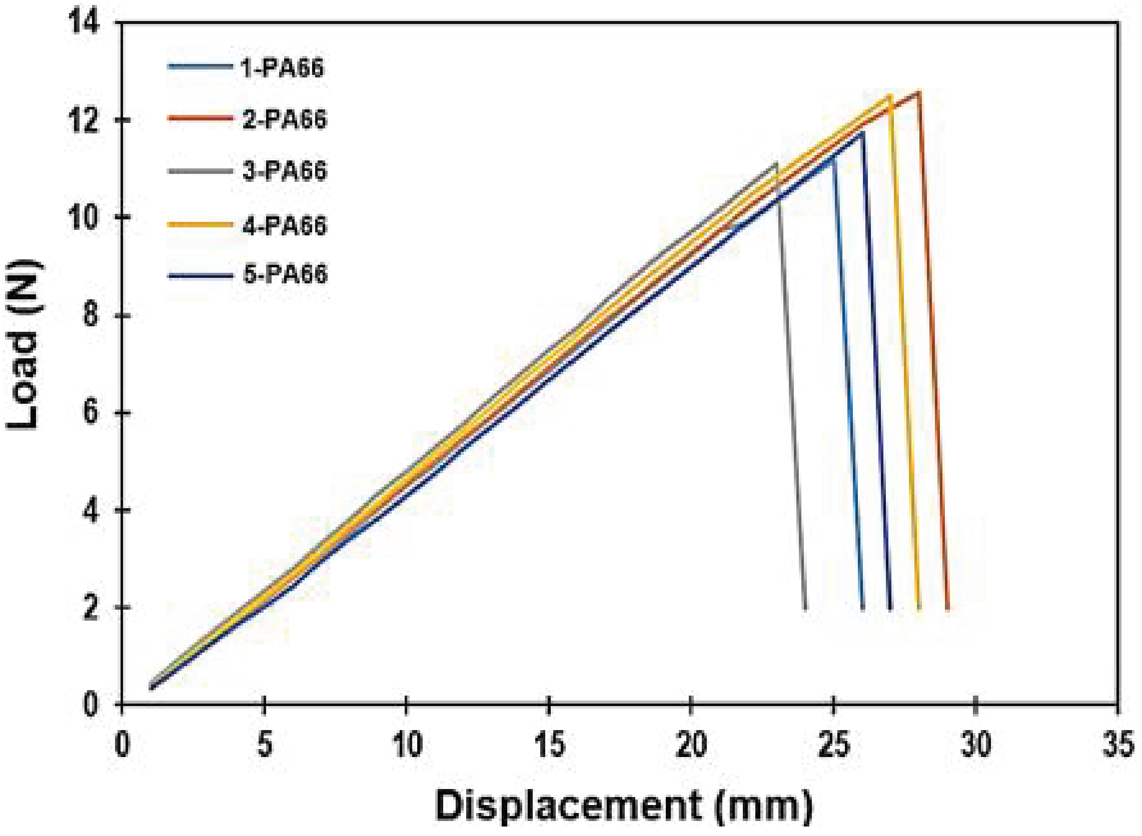


Figure 4.8. Load-displacement graph for electrospinning specimens

The area of the lap joint was used to calculate the results of a single lap shear test as the shear strength. Table 4.2 shows the maximum shear strengths for each sample along with their standard deviations and means, which are also visually presented in Figure 4.9.

Table 4.2. Single lap shear test results for all specimens

Sample Group **Sample No**	**Shear Strength (MPa)**			
	Reference	**L1**	**L2**	**Electrospinning**
Sample 1	9.97	19.59	18.09	17.47
Sample 2	11.68	20.40	19.45	18.30
Sample 3	11.16	20.29	19.69	17.45
Sample 4	11.25	22.00	19.78	20.06
Sample 5	11.21	19.66	19.03	18.78
Average	**11.05**	**20.39**	**19.21**	**18.41**
Standard Deviation (±)	0.64	0.97	0.69	1.17

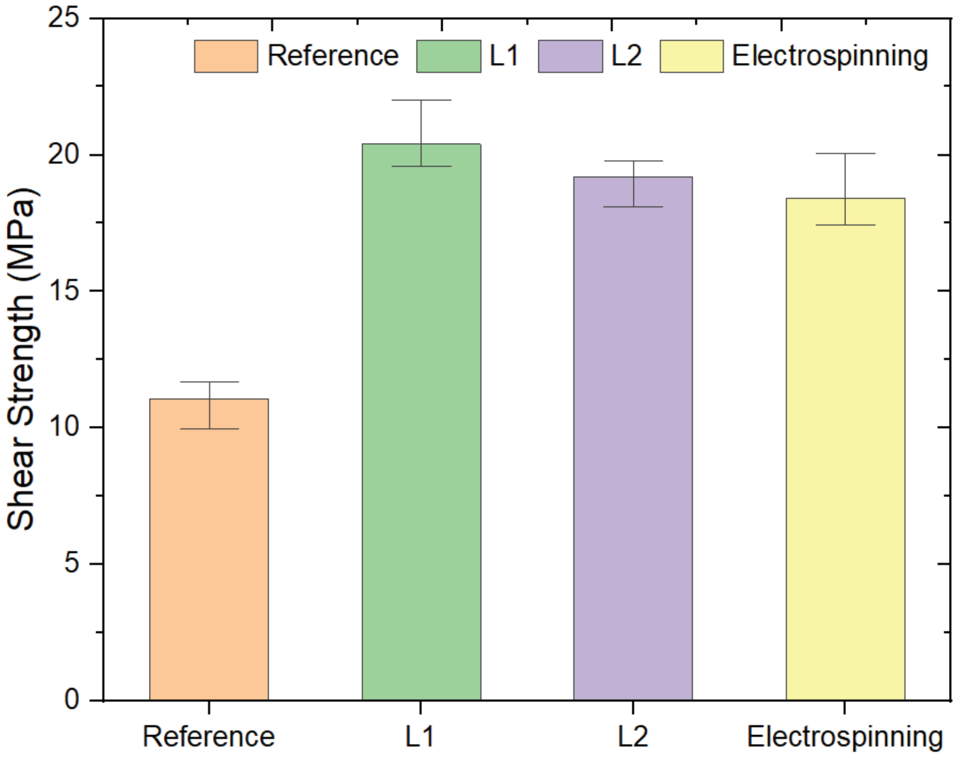


Figure 4.9. Single lap shear test results for all specimens

As a result of the single lap shear tests for all groups represented, the application of surface treatment, both laser surface treatment and electrospinning, had a positive effect on the shear strength of adhesive bonded composite specimens. Figure 4.9 summarises the effect of surface treatment on shear strength for all groups to facilitate comparison.

4.2. Fatigue Tests

Establishment of the fatigue life of adhesive bonding between composite laminates manufactured with UD prepreg plies, single lap shear specimens were prepared. Tensile-tensile fatigue tests were performed using the special test standard of TAI. The effect of applying adhesion surface treatment on fatigue life was investigated in three main groups. The study involved three groups of samples: reference samples without surface treatment, samples treated with laser on the adhesion surface, and samples reinforced with nanofibers using the electrospinning technique on the adhesion surface. Tensile-tensile fatigue tests were conducted on each group using 30%, 40%, and 50% of the average maximum single lap shear loads.

The load-number of cycle curves (S-N curves) present the fatigue test results for each group of specimens. Additionally, stiffness degradation curves were plotted for all groups to observe the stages of fatigue. Tables and graphs illustrate the number of life cycle results for each specimen, along with their mean life cycle. SEM analysis was conducted on fracture surfaces of each group of specimens. The effect of surface treatment on adhesion surface was determined based on SEM images.

Bezazi et al. (2003) mention that checking the stiffness degradation of specimens is a useful method for analysing the start and initiation of damage during fatigue loading. Stiffness degradation curves were plotted to compare and analyse the effect of applying surface treatment on the adhesion surface on fatigue performance. Displacement and cycle values obtained from tension-tension fatigue tests were used to plot the stiffness degradation curves. The initial maximum displacement (d_0) was divided by the maximum displacements for different fatigue cycles (d) to calculate degradation.[43]

Figure 4.10 illustrates the stiffness degradation curve for a reference specimen. The example curve exhibits three main stages, similar to stiffness degradation curves found in the literature. Bezazi and his colleagues explain the reason for these three stages as follows. The first stage shows a sharp decrease in stiffness due to matrix cracking. In the second stage, stiffness degradation progresses slowly due to the progression of cracking that occurred in the first region. In the final stage, stiffness degradation occurs more suddenly than even in the initial stage. This is due to the failure of the sample resulting from fiber breakage.[43]

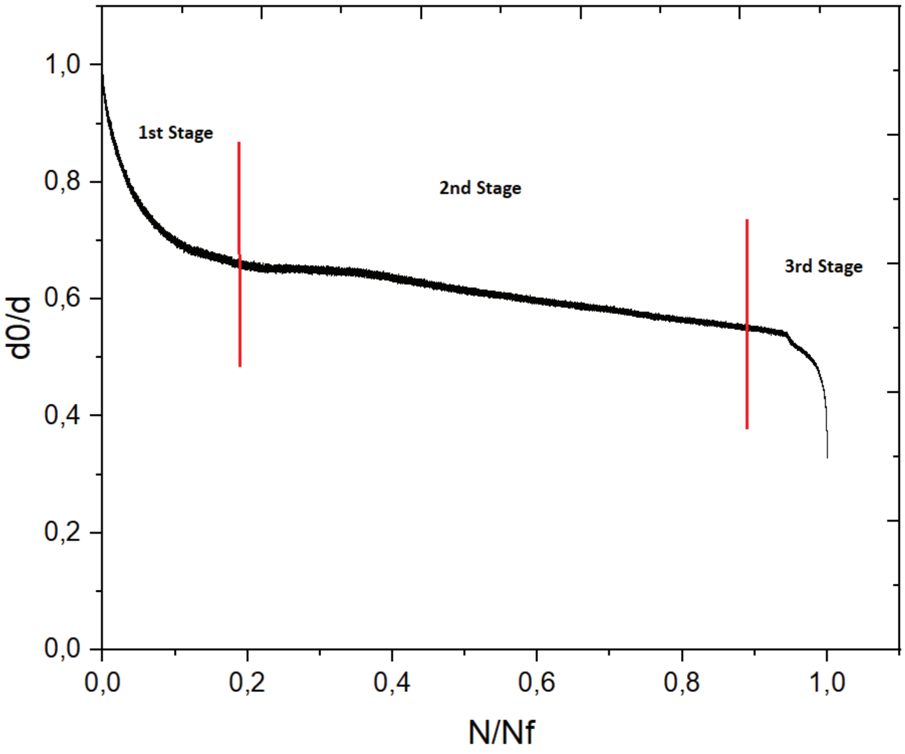


Figure 4.10. Example of a stiffness degradation curve for a reference specimen

Figure 4.11, 4.12, 4.13 and 4.14 display the stiffness degradation curves of reference, L1, L2 and electrospinning specimens resulting from fatigue tests. The x-axis represents the logarithmic number of life cycles, as commonly used in this field of study. The fatigue tests were conducted on each group of specimens using 30%, 40%, and 50% of their average single lap shear load, as illustrated in the figures.

Figure 4.15 illustrates the Wöhler curves for each specimen group. The failure number of cycles, according to the applied percentage of ultimate lap shear strength, was used to plot each curve. The fatigue life was reduced with increasing applied stress level for each group, as expected. For the same applied stress level, the reference specimens had better results than the other specimen groups that had treatment applied to the adhesion surface. For instance, the reference specimens were able to survive up to 10^6 cycles at 30% of their ultimate lap shear strength. In contrast, the L1, L2, and electrospinning specimens could only survive up to 10^5 cycles at the same stress level.

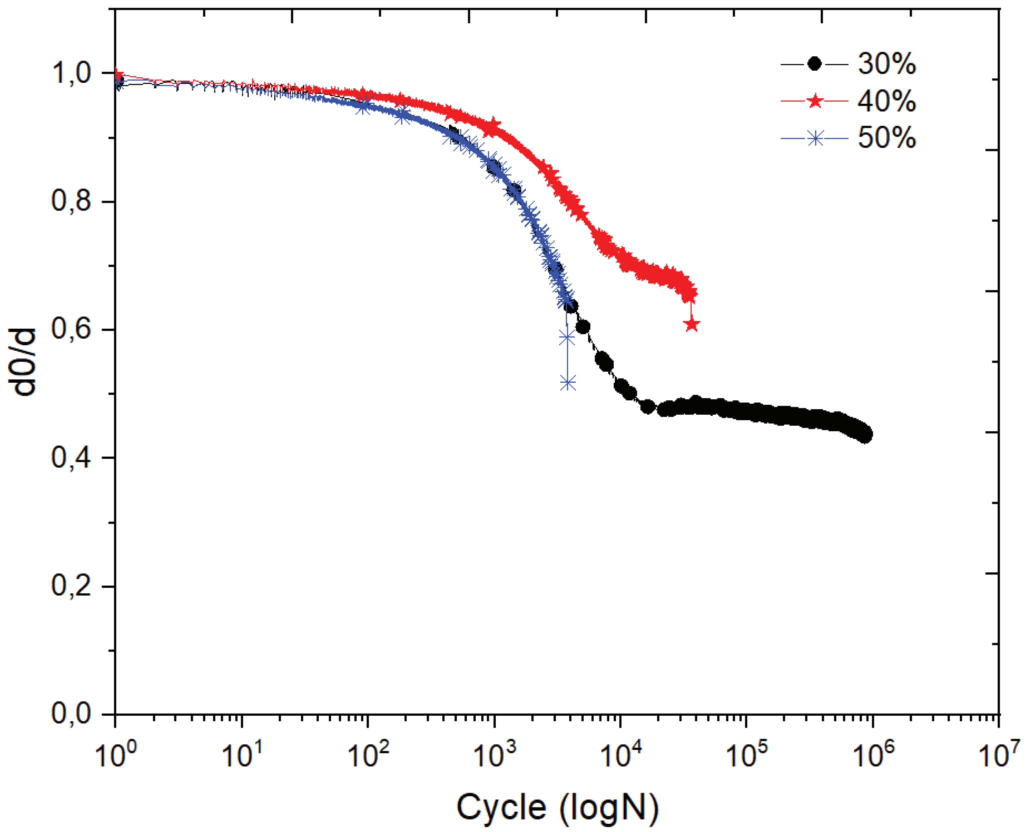


Figure 4.11. Stiffness degradation curves for reference specimens

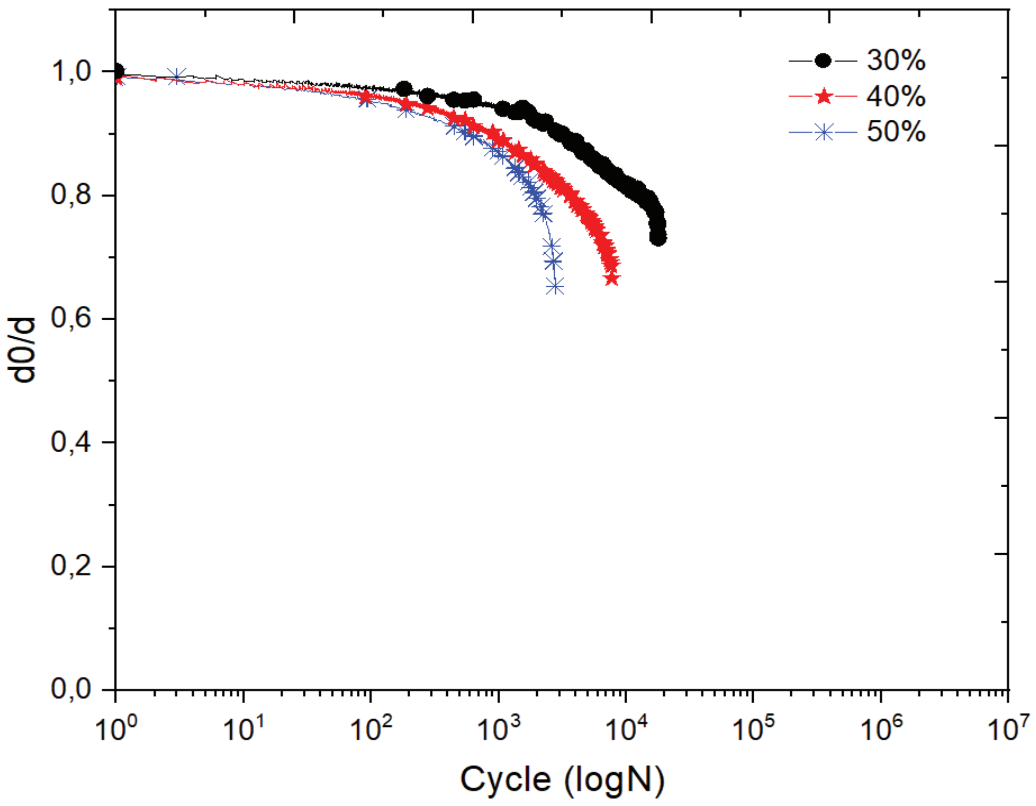


Figure 4.12. Stiffness degradation curves for L1 specimens

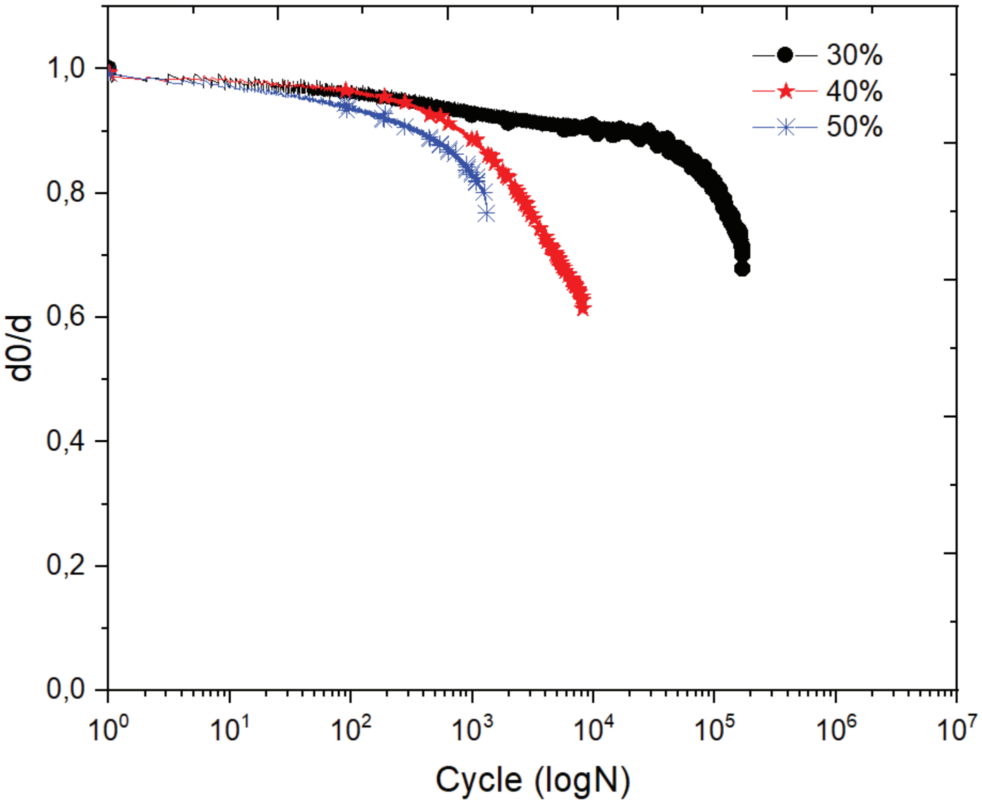


Figure 4.13. Stiffness degradation curves for L2 specimens

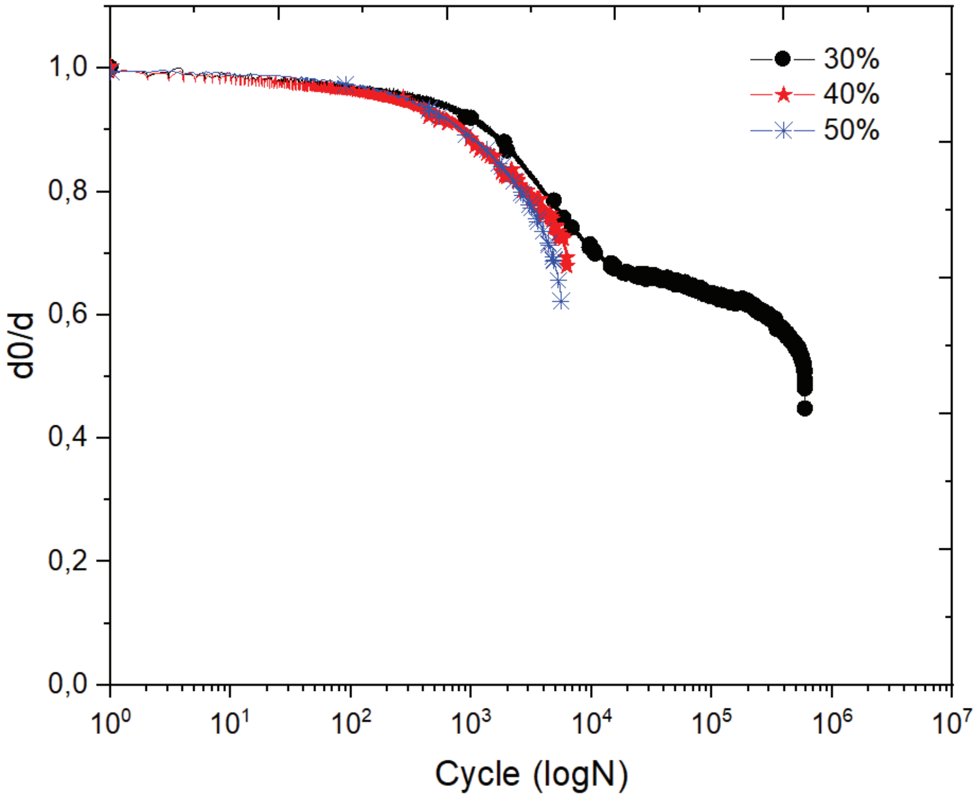


Figure 4.14. Stiffness degradation curves for electrospinning specimens

Table 4.3. Fatigue test results for reference and electrospinning specimens

	Reference Specimens			Electrospinning Specimens		
Percentage of Loads	30%	40%	50%	30%	40%	50%
Applied Loads (N)	1972,03	2629,37	3286,71	3563,22	4750,96	5938,7
Number of Failure Cycles	1718000	78663	8493	900179	77440	5600
	850000	36590	9233	593946	12175	6176
	1132000	65844	3804	925203	6239	7112
Average Cycles	1233333	60366	7177	806443	31951	6296

Table 4.4. Fatigue test results for L1 and L2 specimens

	L1 Specimens			L2 Specimens		
Percentage of Loads	30%	40%	50%	30%	40%	50%
Applied Loads (N)	3946,44	5261,92	6577,41	3718,06	4957,41	6196,76
Number of Failure Cycles	153554	7732	3127	48396	25863	2885
	178445	14283	2782	230687	27079	1311
	180712	16483	4217	169132	8165	2330
Average Cycles	170904	12833	3375	149405	20369	2175

Furthermore, the slopes of the S-N curves for L1, and L2 are very similar and higher than the slope of the curve for the reference specimens. The difference in slope between the curves can be attributed to the failure mechanism of the specimens under fatigue loading.

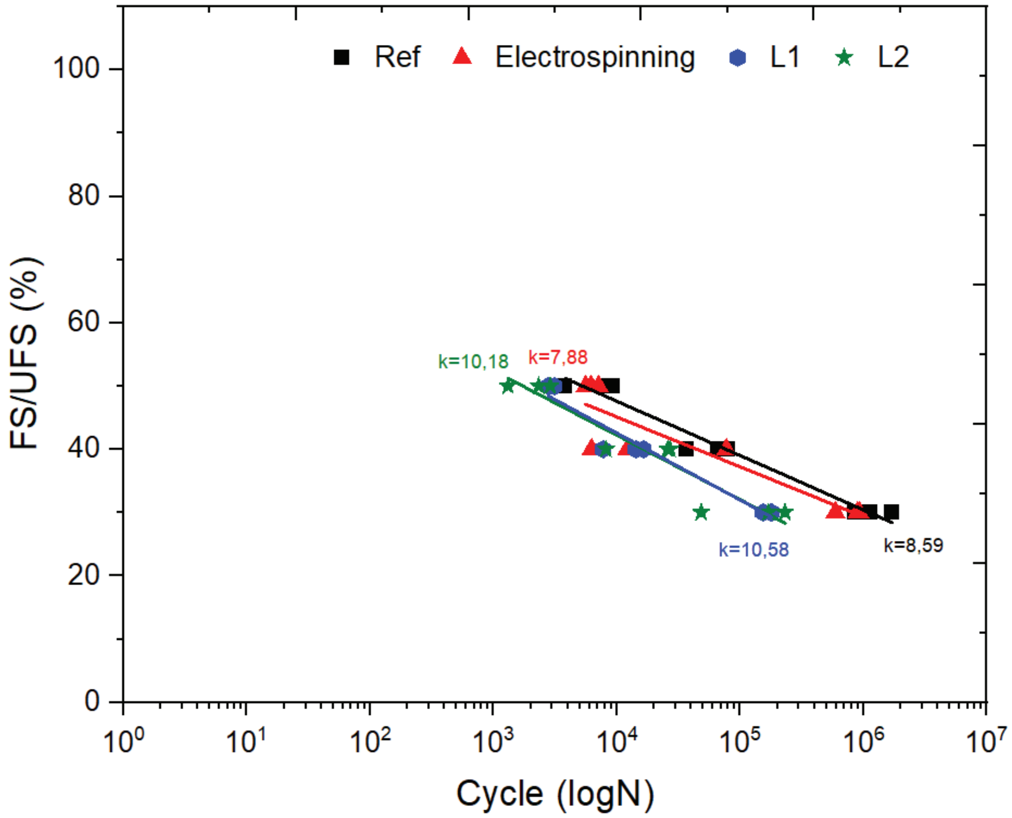


Figure 4.15. S-N curves for all specimens

The aim of the test was to achieve a higher fatigue life for specimens that underwent treatment on the adhesion surface compared to the reference specimens. However, despite these expectations, a decrease in fatigue life was observed for each group that underwent surface treatment, as shown in Figure 4.15. In the literature, Park et al. discussed the effects of surface treatment application. The study illustrate that surface energy and wettability increased with the application of surface treatment as shown in figure 4.19 from the study of İplikçi.[41] However, this did not result in an improvement in mechanical properties, such as fatigue life of specimens. The failure mechanism of the specimens changed from adhesive failure to cohesive failure when the failure surface was analysed. This change in the failure mechanism indicates an improvement in the quality of bonding on the surface.[36]

Changes in failure mechanism were observed in specimens that underwent treatment on the adhesion surface compared to the reference specimens similar to the study conducted by Park and their colleagues. This was determined through SEM analysis of the failure surfaces at IZTECH Center for Materials Research (MAM). SEM analysis images of the fracture surface of the reference, L1, L2, and electrospinning specimens are shown in Figure 4.18. Adhesive failure was observed in the reference specimens (Figure 4.18 a, and b), while SEM images of L1, L2, and electrospinning specimens revealed a change in failure mechanism from adhesive failure to light fiber tear failure.

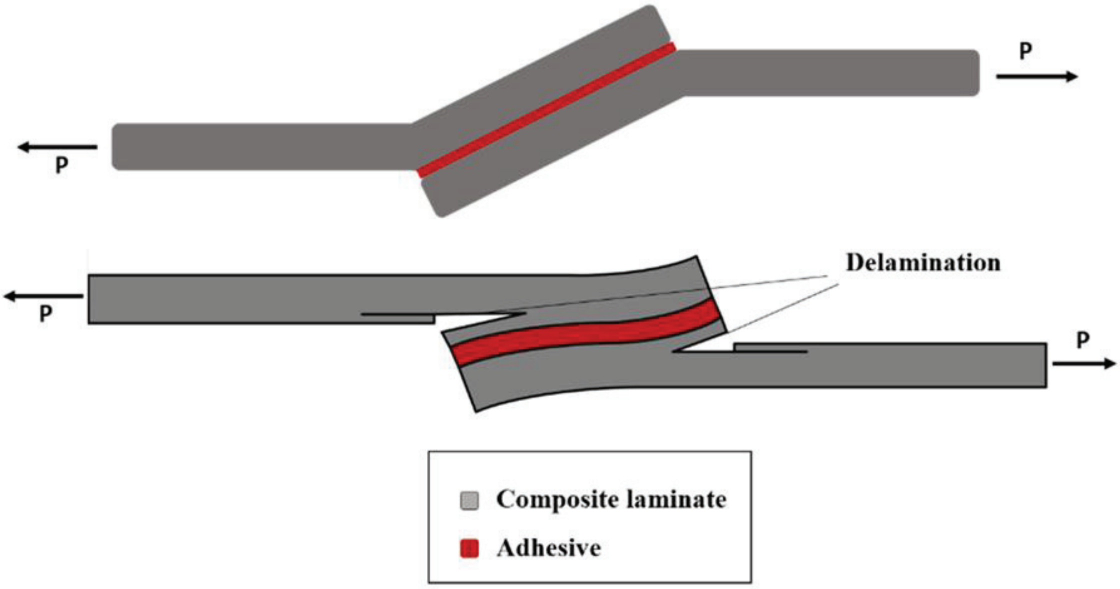


Figure 4.16. The effect of peel stress on delamination
(Source: Malekinejad, H., et al., 2023)

The failure mechanism of specimens that underwent treatment on the adhesion surface was investigated at both macro and micro scales, as shown in Figure 4.21. The adhesion quality between the CFRP composite laminates of the surface-treated specimens was improved compared to the reference specimens. However, the cohesive failure as a failure mechanism that was targeted to be achieved through the applied treatment on the adhesion surface, as described in the literature, could not be achieved for adhesively bonded polymer-based composites after the surface treatment application.

The literature identifies overlap length, adhesive thickness, overlap geometry and corner geometry as key parameters influencing the fatigue behaviour of composite adhesive joints. It has been demonstrated that an increase in overlap length and adhesive thickness has a detrimental effect on the fatigue performance of adhesively joint composites. The experimental results of Mazumdar demonstrate that peel stress increases with increasing adhesive thickness.[44] This increase in peel stress has a significant impact on the composite adherends at the ends of the overlap. Consequently, there is an elevated probability of delamination in adhesively bonded joints, as illustrated in Figure 4.16.[45]

Several numbers of studies have been conducted to investigate the impact of stacking sequence orientation on the fatigue behaviour of polymer-based composites. In these studies, it was observed that the fatigue strength was almost identical when 0° and ±45° plies were used at the interface layer at the joint. Conversely, the use of 0° and 45° plies at the interface layer has been observed to have a differential effect on crack initiation and propagation. When 0° plies are utilised at the bonding interface, debonding occurs on the adhesive layer and subsequently propagates along it as a consequence of the adhesive layer's cohesive failure. Conversely, when 45° plies are utilised at the bonding interface, debonding commences at the adhesive layer and subsequently propagates through the ± 45° plies of the adherend, accompanied by delamination. The crack growth on the adherend layers ultimately reaches 0° ply, at which point the final failure occurs. The influence of layer orientation at the bonding interface on crack initiation and propagation is clearly demonstrated in Figure 4.17.[45]

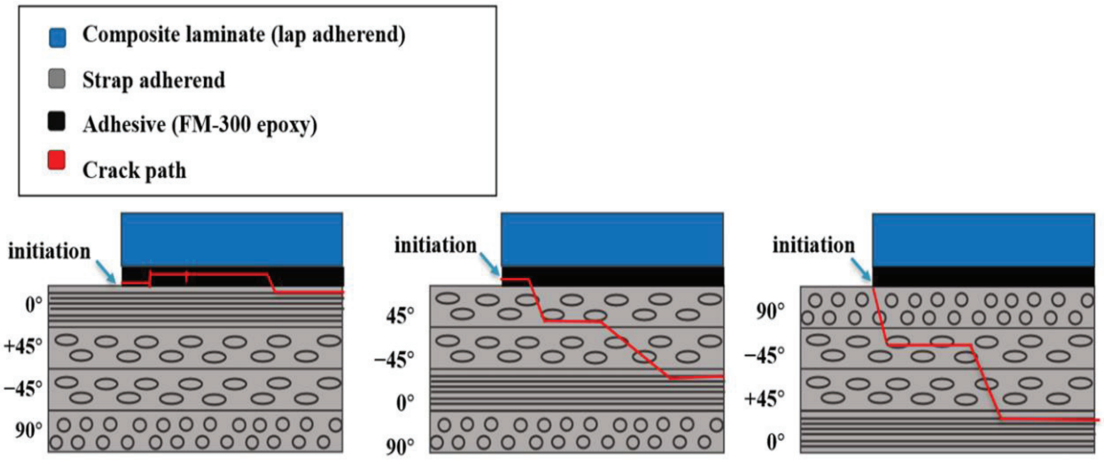


Figure 4.17. The effect of layer orientation at the bonding interface on the crack initiation and propagation (Source: Malekinejad, H., et al., 2023)

Furthermore, existing research indicates that the sequence in which components are stacked has a significant impact on the fatigue performance of adhesively bonded composite joints. In a study of the literature, all 0° ply specimens exhibited an increased fatigue strength of 30%. Also, in a separate study of the literature, the utilisation of a full

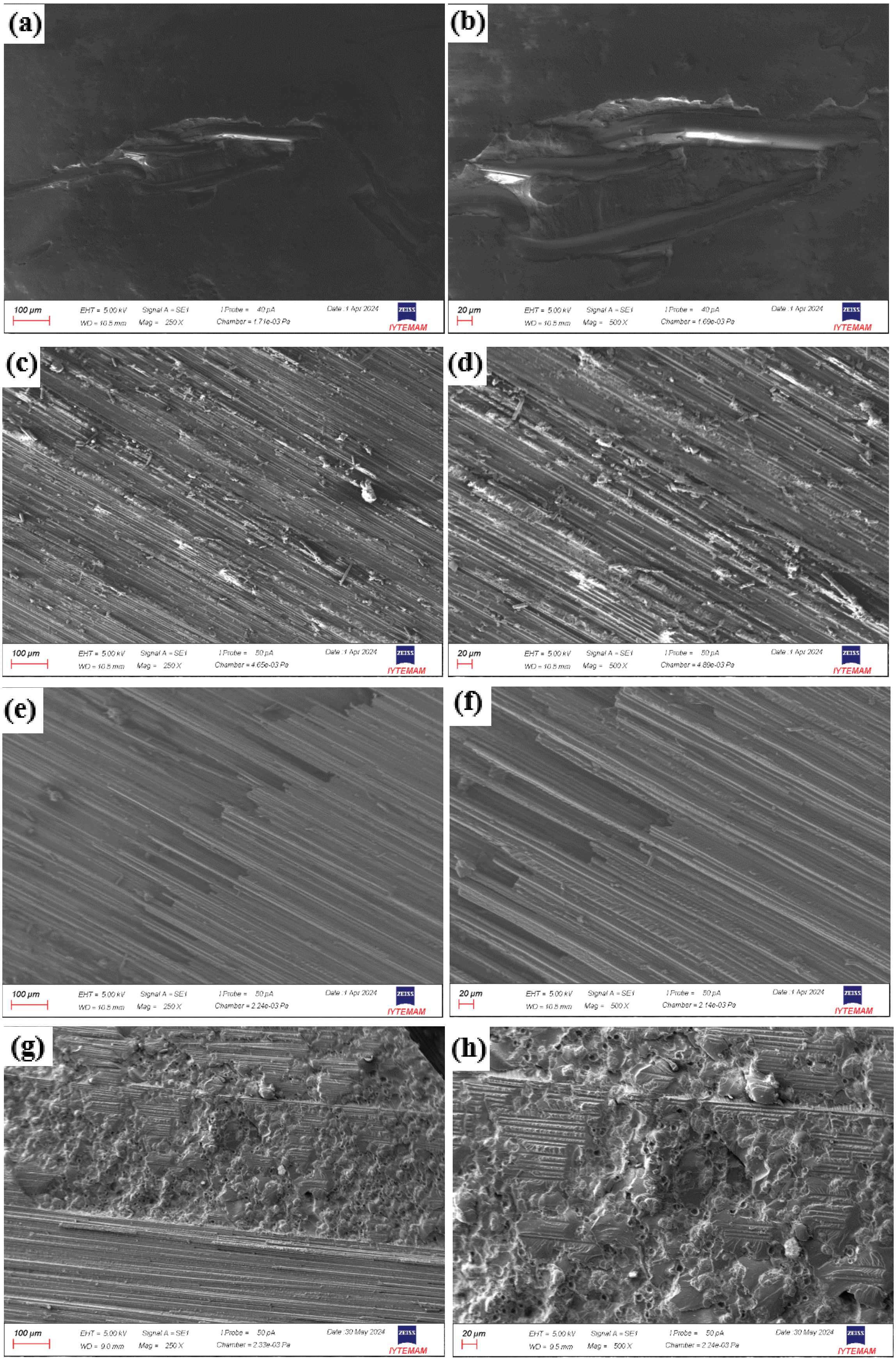


Figure 4.18. SEM images of fracture surface of (a-b) reference specimens, (c-d) L1 specimens, (e-f) L2 specimens, (g-h) electrospinning specimens

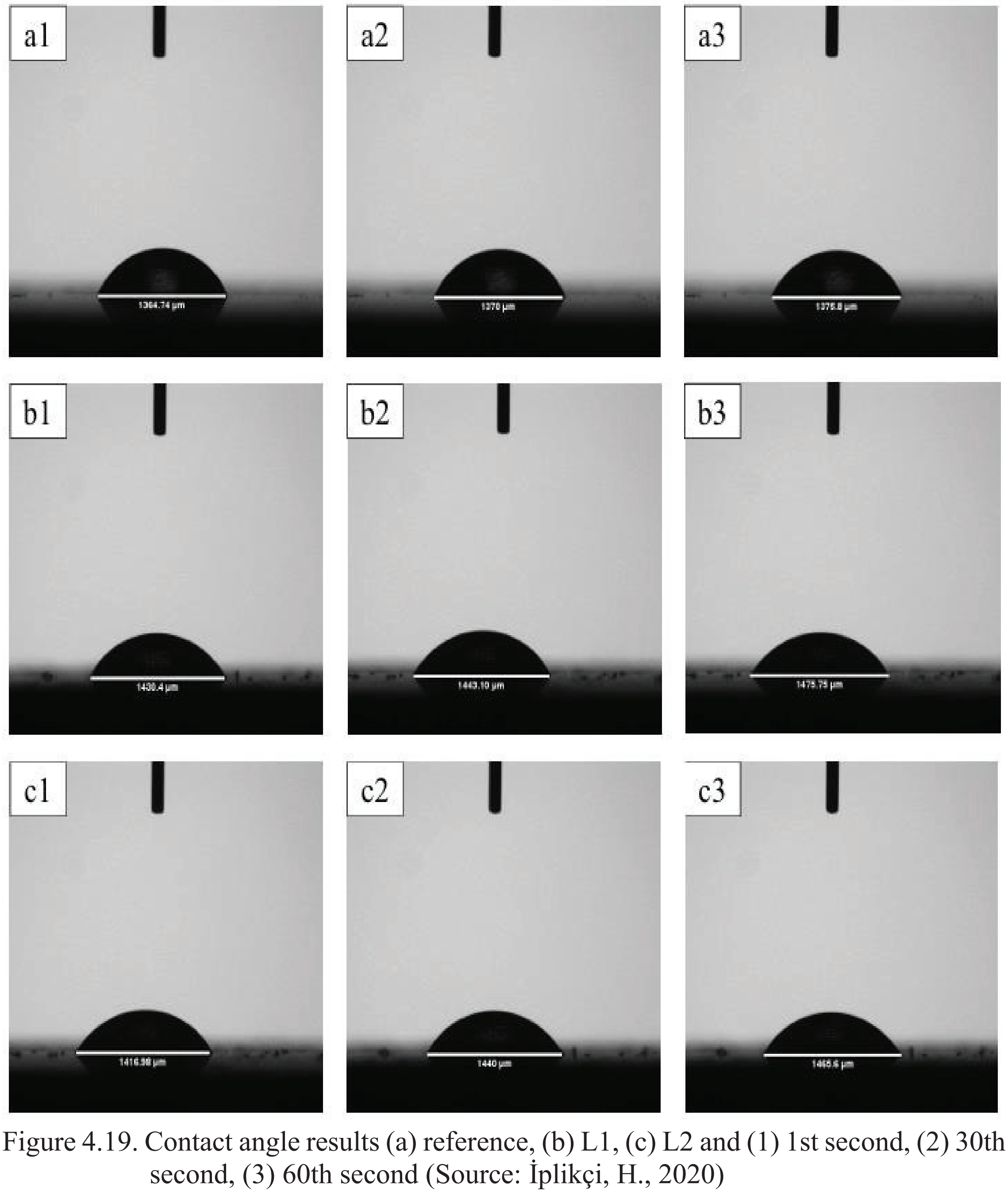


Figure 4.19. Contact angle results (a) reference, (b) L1, (c) L2 and (1) 1st second, (2) 30th second, (3) 60th second (Source: İplikçi, H., 2020)

0° ply in a stacking sequence was found to result in the occurrence of cohesive failure. Conversely, when a 45° ply was utilised in conjunction with the adhesive layer, the crack path and failure mechanism were observed to occur within the adherend. The results of these studies indicate that the proportion of 0° ply has a direct effect on fatigue strength and also on the failure mechanism. Figure 4.20 illustrates the effect of the number of 0-degree plies on the fatigue strength of the composite material.[45]

Figures 4.18 and 4.21 illustrate scanning electron microscopy (SEM) images of the fracture surface of specimens that underwent surface treatment. In these figures, the transition from adhesive failure to fibre tear failure in the failure mechanism was investigated with the application of surface treatment. The observed change in failure mechanism indicates that the adhesion quality was enhanced by the surface treatment on the adhesion surface. However, the results were not entirely satisfactory due to the inability to reach the cohesive failure point. As previously stated, the stacking sequence of adherends, in particular the orientation of the layers at the bonding interface, may be the underlying causes of delamination. As previously documented in the literature, the aforementioned delamination resulted in the occurrence of fibre tear failure as a failure mechanism.

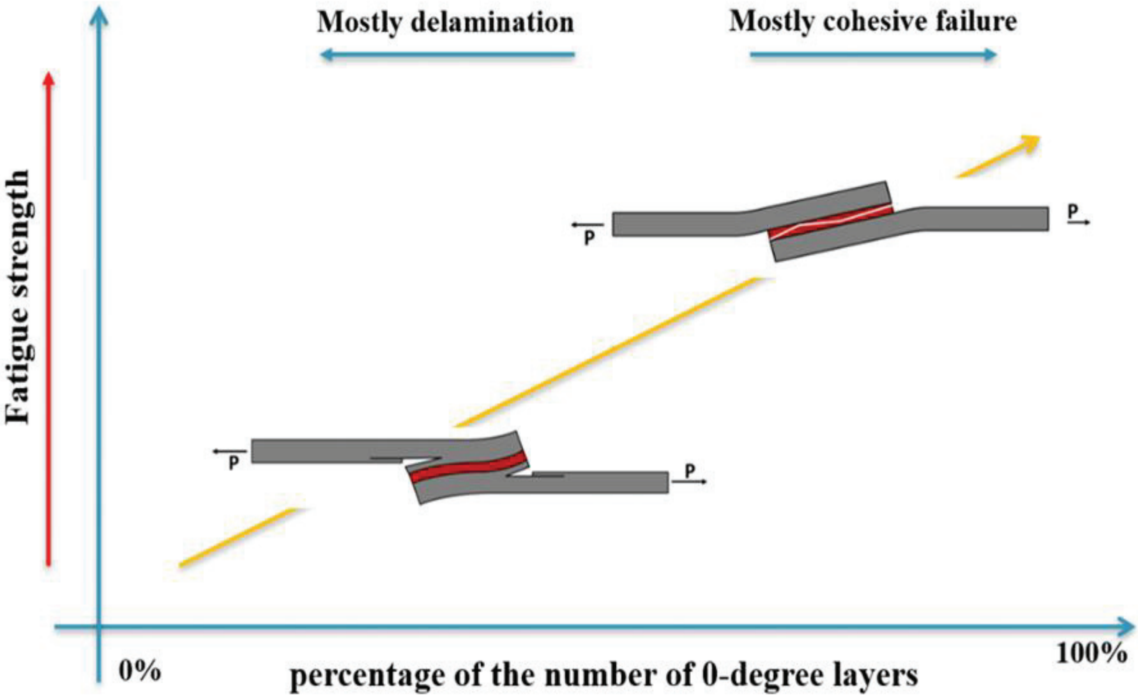


Figure 4.20. The effect of the number of 0-degree plies on the fatigue strength of the composite material (Source: Malekinejad, H., et al., 2023)

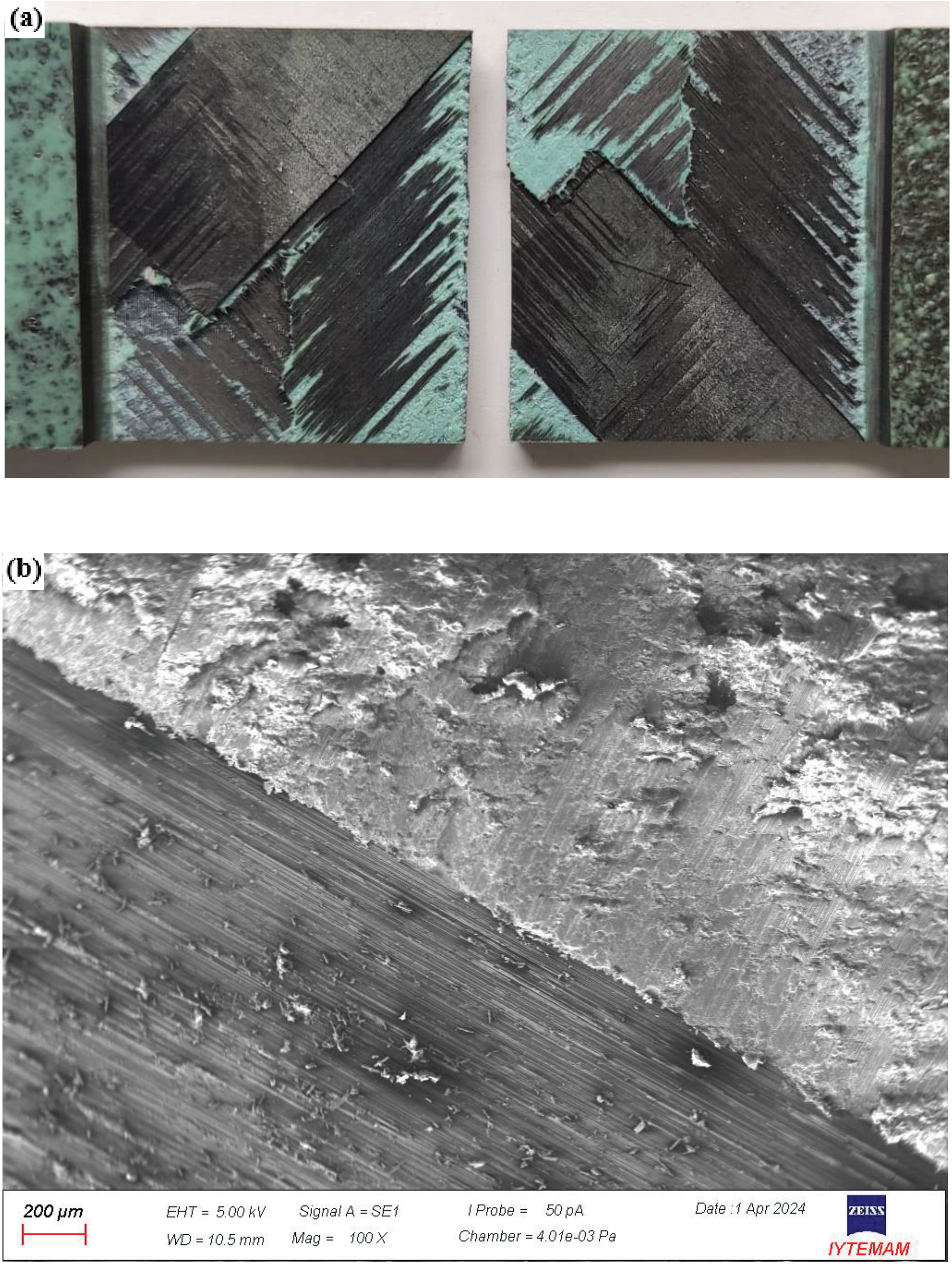


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Figure 4.21. Fracture surface of specimens with (a-c-e) macro and (b-d-f) micro scale images

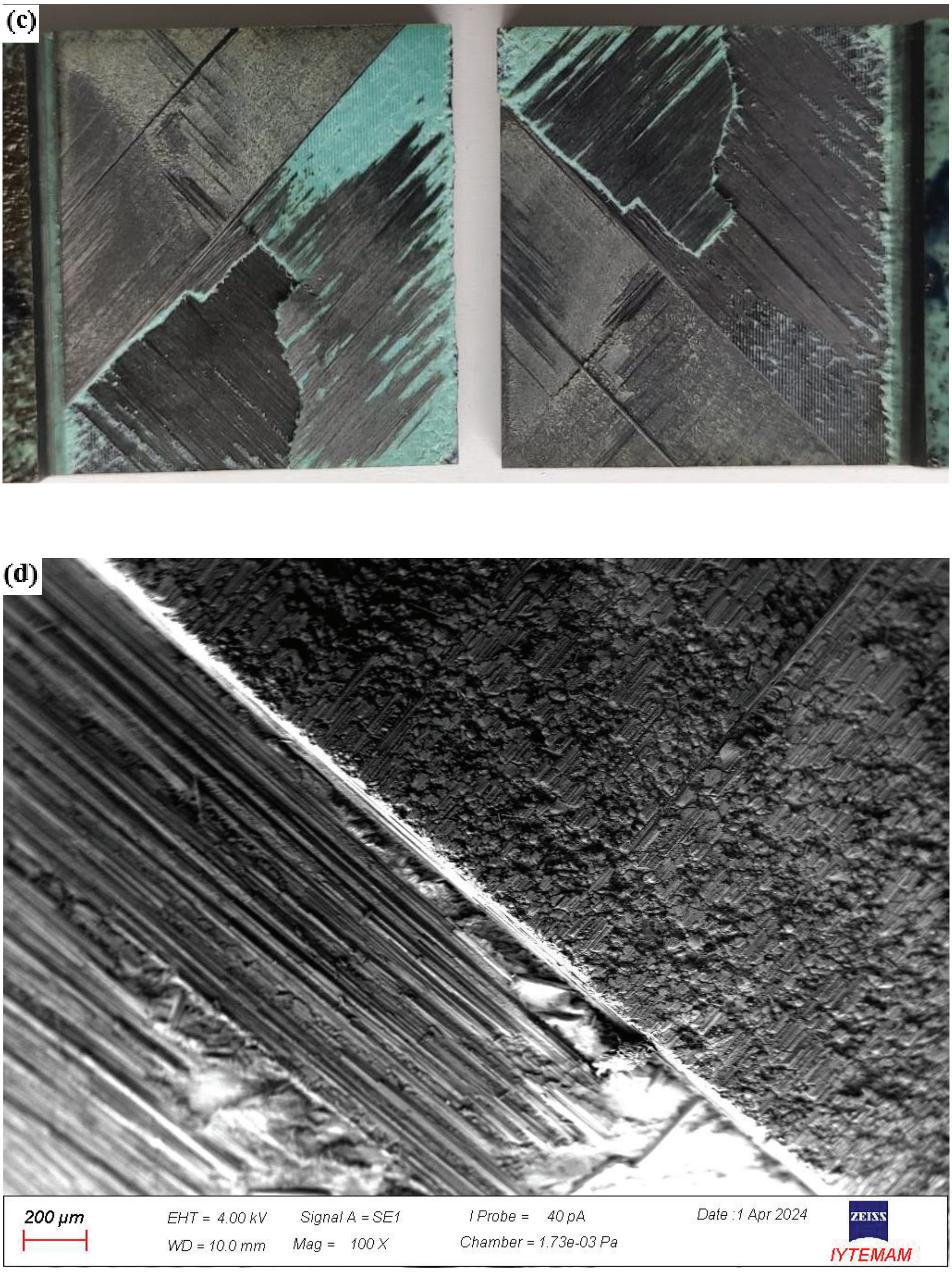

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Figure 4.21. (cont.)

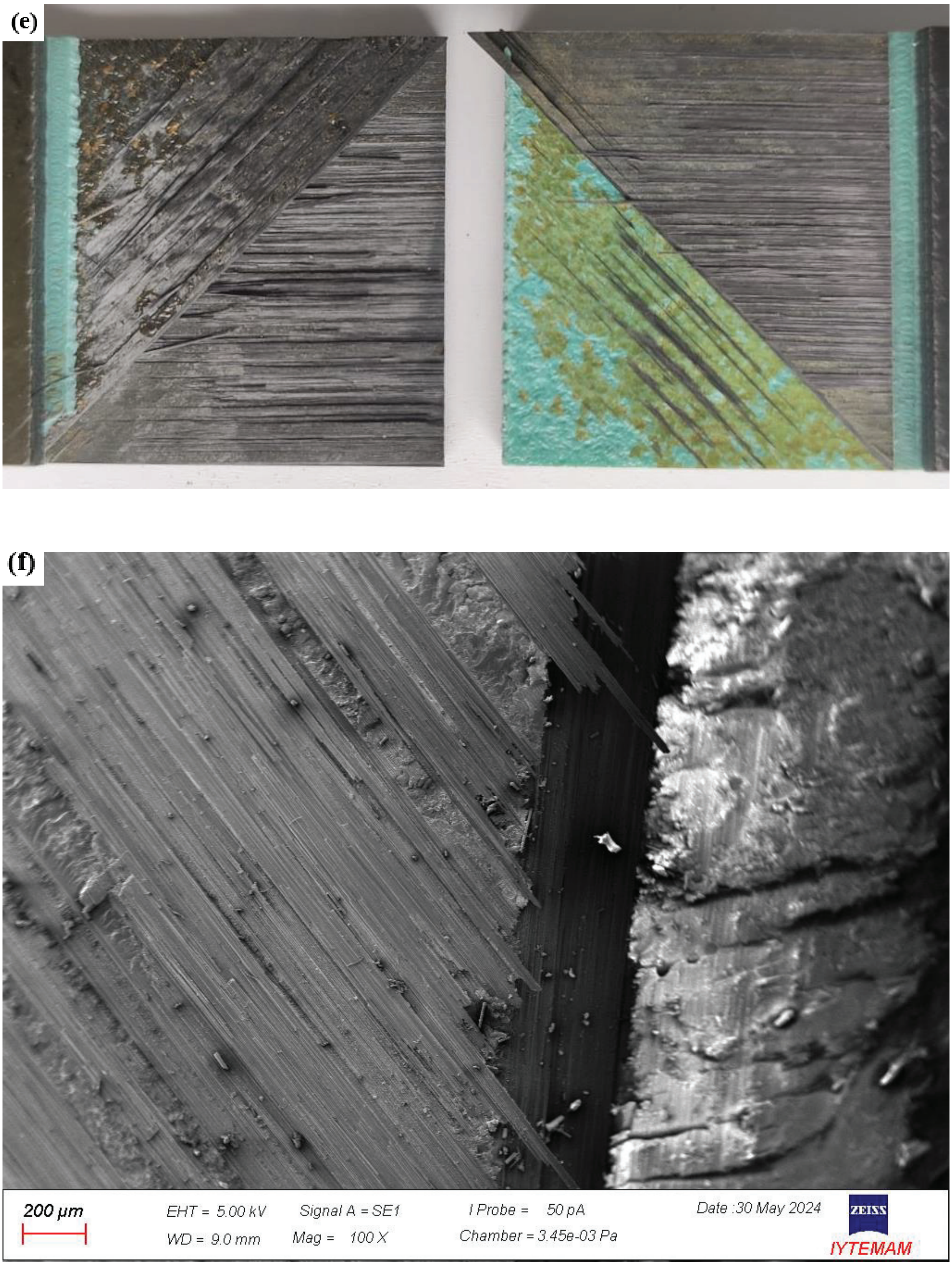

Figure 4.21. (cont.)

CHAPTER 5

CONCLUSIONS

Fiber-reinforced composite materials have been employed in a number of industries that require the use of high-performance and lightweight materials, including defence, aviation, high-performance automobiles, and others. In addition, prepregs, which reduce the amount of residual resin and facilitate production, are becoming increasingly popular in most industries. The performance of composite materials is contingent upon a number of parameters, including the reinforcement type, matrix material, fibre arrangement, and the interface between the fibre and matrix. In the current industrial climate, there is a demand for composite materials with enhanced performance. In response to these industry needs, research efforts have been redirected towards this particular area. Nanomaterials are increasingly being employed as reinforcements with the objective of enhancing the mechanical properties of composites. Electrospinning is a highly effective and readily applicable technique for the production of nanofibers, which can be utilised as reinforcing materials in composites. Nanofibers, which are produced by the electrospinning technique, are generally dispersed throughout the uncured prepregs, and this does not result in a significant increase in the thickness of the laminates.

The majority of products are not manufactured as a single piece; at least one joining method must be employed for the joint parts. A variety of joining methods are employed in the industrial sector. One of the most prevalent joining techniques is undoubtedly mechanical fasteners, which may be considered a traditional joining method. It is well established that composite materials are not isotropic, which means that drilling processes, which are typically required for the use of mechanical fasteners, can result in delamination. This phenomenon results in the detection of mechanical properties that are lower than expected in composite materials. In the present era, adhesive bonding is a particularly favoured method, particularly in the context of composite materials. This is due to a number of advantages, including a reduction in fuel consumption as a result of weight reduction, a reduction in radar absorption, and a reduction in stress concentration, in comparison to traditional joining techniques. Some industries, including the aviation and defence sectors, are seeking to achieve superior adhesion quality and mechanical properties in adhesively bonded joints. Some researches have indicated that the application of surface treatments, such as laser treatment, to the bonding surface can positively affect the adhesion quality, surface energy, wettability, and mechanical properties of the bonded joint.

The objective of this study is to investigate the effect of surface treatment application on the fatigue performance of adhesively bonded polymer based composites. The electrospinning method and laser surface treatment were selected as the treatment methods to be applied to the bonding interface. In the case of the electrospinning method, the PA66 nanofibers were coated onto the CF/EP prepregs. The rationale behind the selection of PA66 as a nanofiber is its solubility in a majority of solvents, relative affordability, negligible thickness increases after curing, and superior mechanical properties compared to other polymers. Conversely, in the case of laser treatment, a nanosecond pulsed fiber laser was employed. The parameters were selected to ensure that the epoxy was removed from the surface without any damage to the fibers. Two different offset distances were employed in the laser treatment of the CFRP laminates, namely 0.15 mm and 0.20 mm. The laminates were produced using CF/EP prepregs.

In the initial stages of production, CFRP plates were manufactured using CFRP laminates, which were produced with CF/EP prepregs. The plates were then bonded together with a 3-layer (0.6 mm) FM300K film adhesive using an autoclave technique. Single lap shear tests were conducted on four groups of specimens, namely the reference group, the 0.15 mm laser offset distance group (referred to as L1), the 0.20 mm laser offset distance group (referred to as L2), and the electrospinning group. The results of the single lap shear tests demonstrated that surface treatment of the bonding interface enhanced the mechanical properties of polymer-based composites, regardless of the laser treatment or electrospinning techniques employed. Tension-tension fatigue tests were conducted on each group of specimens, based on the average maximum shear strength value of each specimen. The dimensions of the fatigue test specimens were prepared in accordance with the specifications set forth in ASTM D5868, and the fatigue tests were conducted in accordance with the specifications set forth in the TAI standard.

The a priori expectation was that the application of surface treatment to the bonding interface would result in an improvement in fatigue performance. The results of the tests indicated a significant reduction in fatigue life for the specimens. In the case of laser surface treatment (for both L1 and L2), a 75% decrease in fatigue life cycles was observed in comparison to the reference specimens. In the case of the electrospinning method, a 35% decrease was observed.

Conversely, when fracture surfaces were investigated for specimens, it was observed that the surface treatment applied to the adhesion surface resulted in superior adhesion quality compared to the reference. According to the literature, cohesive failure is the desired failure type to be achieved as a result of surface treatment application for improved adhesion quality. In this study, when fracture surfaces were investigated in both macro and micro scales, it was found that the failure mechanism changed from adhesion failure to fiber tear failure as a result of surface treatment application. The current stacking sequence in the bonding interface was identified as the primary factor contributing to the observed fiber tear failure mechanism and significant reduction in fatigue life cycles indirectly.

5.1. Future Works

- The investigation will examine the influence of different adhesive types and spew fillet effects on fatigue performance.
- The impact of varying stacking sequences on fatigue performance will be investigated.
- The fatigue performance of carbon fibre-reinforced polymer (CFRP) plates that have been subjected to ageing will be investigated and compared with that of non-aging specimens.
- A numerical analysis will be conducted in order to make life estimation, with the results being compared with those obtained from experimental procedures.
- Lamination and the production of test specimens will be conducted using a hot press. This process will be used to ascertain the influence of the production method on fatigue performance.

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