



## **An experimental study of effect of atmospheric plasma treatment on shear strength of adhesively bonded GFRP-aluminum joints**

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### **Abstract:**

The objective of this study is to investigate the effect of air (dielectric barrier discharge) DBD plasma treatment on the bonding strength of adhesively bonded glass fiber-reinforced epoxy composite-aluminum lap joints. The bonding performance of lap joints produced by the plasma treatment was compared with that of untreated and peel-ply surface treatments. Water contact angles of the substrates were measured for untreated, peel-ply, and plasma surface-treated substrates. Experimental results showed that plasma-treated aluminum and GFRP substrates increased the wettability properties and thus shear strength of adhesively bonded GFRP-Al joints increased. After the shear tests, the fracture surfaces of the substrates were visually examined and three different damage modes were observed, including light fiber tear failure, adhesive failure, and thin layer cohesive failure modes.

## **1. Introduction**

Composite materials are currently being used in many kinds of industries. The most exciting material that can take the place of conventional components, metals, and woods is polymer matrix composites. Fiber-reinforced polymer matrix composites, or FRPCs, have gained increasing use in a wide range of industrial, automotive, engineering, and structural applications [1]. The demand for lightweight with high strength materials has led to the use of fiber-reinforced composites in engineering applications [2]. The major load-bearing component is composed of fibers, which are surrounded by the matrix, and the matrix acts as protection in the intended direction. As the fibers come from materials like glass, carbon, or aramid (kevlar), the composition of the matrix in fiber-reinforced polymers may differ.

Some thermoset polymer matrices include epoxy, phenolic, polyester, and vinyl ester [3]. Fiber-reinforced polymer composite offers numerous benefits, including low density, high strength, and ease of manufacturing [4]. Among other fiber-reinforced materials, glass fiber-reinforced polymer (GFRP) and carbon fiber-reinforced polymer (CFRP) composites have started to replace conventional materials due to their low specific weight and higher strength [5]. GFRP composites provide excellent fatigue and have durability against corrosion, minimal cost for maintenance, and the capacity to form complex designs without a requirement for machining [6]. GFRP is less expensive in comparison to other varieties of fiber-reinforced polymers. Nonetheless, due to its low elastic modulus, GFRP may not always provide sufficient rigidity to the structure [7]. A complete composite solution might not be feasible in many

applications on a large scale since composites need to be joined with metals. Effective methods of joining are consequently necessary for the reliable usage of composite materials. The improvement of effective methods for joining metals and composites can be considered an essential requirement for increased composite applications [8]. The proper joining technique must be used to ensure a safe transfer of load at a low weight. As a result, forming dissimilar, high-strength connections between metal and fibre-reinforced polymer components poses a major challenge for joining methods and remains a crucial focus of research [9].

Today, many different methods are used to join composite and metal parts. Mechanical fastening, one of the traditional techniques, is used to mechanically join two or more parts [10]. In this technique, a third component, such as a bolt or rivet, is used to join the different parts [11]. One of the traditional methods is mechanical fastening, where two or more parts are joined mechanically using a third component like a bolt or rivet. This method is popular due to its simplicity and the ease of disassembling the joined elements. On the other hand, when a mechanically fastened joint is exposed to load, undesired stress accumulation causes micro-level damage in the joined components and opened holes, which degrades the structural integrity of the joint. Adhesive joints can be utilized instead of conventional joining techniques to solve these problems [10]. Adhesive bonding is defined as the joining of the surfaces of the adherends using an adhesive. In engineering applications, adhesively bonded joints are an alternative to mechanical joints and have numerous benefits over traditional mechanical fasteners [12]. An easy and effective way to join structural parts without drilling is adhesive bonding [13]. In recent years, particularly adhesive bonding applications of metal to composite parts have increased in automotive, boat structures, marine, aerospace, and wind turbine blades [14-17]. Surface pretreatments for adhesive bonding should ensure the removal of all impurities (e.g., lubricants, dirt, weak corrosion layers, etc.) from the areas to be bonded. This is essential to achieve high-strength adhesive joints [18]. Commonly used surface treatments to enhance adhesive bonding performance consist of sanding, grit blasting, peel ply removal, and plasma treatment, among other methods. Dry cleaning processes, such as plasma and laser treatments, offer exciting alternatives to conventional surface treatment methods. Plasma treatment of polymers and metals to improve adhesion has received a lot of attention [19]. Ionized gases can elevate the surface energy of polymers, ionize neutral substances on polymeric surfaces, generate reactive species (such as OH and NH-

bearing groups), activate atoms, and nano-modify the surface. Because of the ionized molecules, an extremely high-energy plasma is produced [20]. Many kinds of plasmas have been used in industry and are produced in research laboratories. Plasmas can be classified primarily based on the power supply that generates them, the atmospheric pressure of the operating gas, the shape of the electrode, and the type of discharge [21]. In recent years, atmospheric (cold) plasma has been the most common approach for surface modification of polymers [22]. It is much cheaper than other plasma processes because it does not require chambers and expensive vacuum devices [23]. Therefore, it provides easier use in large-volume industrial processes [24]. It is well known that dielectric barrier discharges (DBD) are an excellent plasma source for the treatment of surfaces [25]. The surface morphology and chemical composition of polymers are modified by DBD exposure, which also results in changes in wettability and roughness [26]. Several studies have indicated the effective application of cold plasma treatments for the surface modification of aluminum and its alloys.

Rhee et. al. applied DC plasma surface treatment to the aluminum panel and examined its effect on the bonding properties of aluminum-CFRP joints. The optimal plasma parameters were found to be a volume ratio of acetylene gas to nitrogen gas of 5:5 and a treatment time of 30 s [27]. Atmospheric pressure plasma had been used by Saleema et al., to clean aluminum alloy 6061-T6 and increase the durability of adhesive-bonded joints. Under pristine conditions, the plasma achieved a high adhesion strength of  $24 \pm 1$  MPa on surfaces after only 15 s of exposure [28]. In a study by Xun Wang et al., atmospheric pressure plasma processes were applied to aluminum using three different gases (nitrogen, argon, and air) to increase the strength of adhesively bonded aluminum alloys. The contact angle and surface free energy of the treated aluminum were measured. Then, single-lap shear tests of the joints were conducted. The results of the study revealed that air and nitrogen plasma treatments greatly improved the wettability of aluminum specimens, resulting in increased joint strength [29]. The chemical and physical changes created by the DBD method in the ambient air on the surface of three-dimensional woven carbon fiber-reinforced epoxy composites were examined in a study by Hao Li et al.. Contact angle measurements, X-ray photoelectron spectroscopy (XPS), and atomic force microscopy (AFM) analysis were carried out. It was revealed that the surfaces of the plasma-treated samples were more active, hydrophilic, and rougher [26]. In a research by Sorrentino and Carrino, the effect of cold plasma surface treatment on aluminum

surfaces in terms of wettability was examined. It has been shown that treating the aluminum surface with oxygen-cold plasma enhances its wettability and adhesion [30].

In this work, the influence of plasma treatment on the adhesive bonding of GFRP-Al was investigated. The GFRP and aluminum substrates underwent air DBD plasma surface treatment with varying applied voltages and treatment durations. Contact angle measurements of surface-treated samples were performed. Single-lap adhesively bonded joints of composite-aluminum specimens were produced, and shear tests of the joints were carried out to assess their lap shear strength. Then, the failure modes of fractured specimens were determined to gain insights into the impact of plasma treatment on the adhesive bonding process.

## 2. Material and Methods

### 2.1. Materials

GFRP and aluminum alloy 5083 were chosen as the substrates for adhesive joints. Areal density of plain-woven E-glass fiber fabrics was 300 g/m<sup>2</sup>. The glass fiber-reinforced epoxy composites were fabricated by vacuum-assisted resin infusion molding (VARIM). It is a significant method for producing composite materials due to its cost-effectiveness and applicability for large structures. The resin and hardener used in the production of composites were F-1564 epoxy and F-3487 hardener, respectively. Each composite plate was produced using an eight-layer woven glass fiber-reinforced epoxy resin matrix. The final thickness of the GFRP composite was approximately 2 mm. A wet-cutting machine was used to cut the GFRP composite and aluminum alloy into dimensions of 25x100 mm.

Araldite® 2015 was used as an epoxy adhesive to produce single-lap GFRP-Al joints. It is a two-component (resin and hardener) epoxy paste adhesive with high shear and peel strength. Furthermore, it is an ideal adhesive for bonding dissimilar substrates. A fixture was designed to ensure that adhesively bonded single-lap joints have the same bonding surface area, volume, and thickness. Furthermore, equal pressure could be applied to the adhesive tube with a mechanical applicator, and a mixer apparatus was utilized to homogeneously mix the adhesive. Adhesively bonded single lap joints were kept at room temperature for 24 hours and then cured in an oven at 80 °C for 2 hours. The joints were kept at room temperature for 7 days before shear tests were performed.

### 2.2. Surface treatments

The surfaces of all substrates were prepared by cleaning with acetone to remove contaminants before the application of the surface treatments. For the peel-ply treatment, a nylon layer was added to the produced composite material. After curing the composite material, the nylon layer was removed and a rough surface was obtained.

Plasma treatments were applied to GFRP and aluminum specimens using air DBD plasma (PVM500) as shown in Figure 1. All plasma surface treatments were carried out with a discharge gap of 3 mm. In this study, the applied voltage of plasma was set to 30 kV, 40 kV, and 50 kV, and the treatment time of plasma was set to 10 s, 30 s, and 90 s. The optimum plasma parameters to be applied to the GFRP composite sample was selected as 50 kV-90 s based on our previous studies. Three different values of plasma voltage and duration were selected for aluminum and their effects on the mechanical strength of GFRP-Aluminum adhesively bonded joints were investigated.

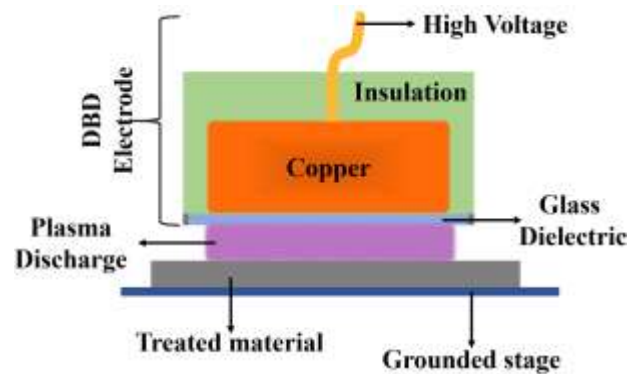


Figure 1. Schematic illustration of the experimental setup for atmospheric plasma surface treatment

### 2.3. Contact Angle Measurements

Water contact angle measurements were carried out to examine the effect of air DBD plasma treatment on wettability. Water contact angles were measured within 5 minutes after applying the plasma treatment to the substrate surfaces. The contact angles were read using graphing software when the test liquid droplet (4 µL) stabilized on the substrate surfaces. Five repeated measurements were performed for each parameter, and the average values were calculated.

### 2.4. Single Lap-Shear Tests

Lap-shear tensile tests of adhesively bonded joints of dissimilar substrates were conducted in accordance with ASTM D5868 standards, as shown in Figure 2. Each joint was tested on a universal testing machine

(Shimadzu Autograph AG-IS, Japan) at a crosshead speed of 1.3 mm/min. To reduce the effects of bending stresses during shear tests, end tabs of the same thickness were bonded to either substrate. Five adhesively bonded joint samples were prepared for each examined parameter in accordance with the test standard, and the average of the shear strength results was calculated. Joint strengths reported as shear stress were obtained by the following equation 1:

$$\tau = \frac{P}{A} \quad (1)$$

Where P is the maximum load and A is the overlap area.

After the single-lap shear tests, failure analysis was conducted for the fractured surfaces of all tested joints.

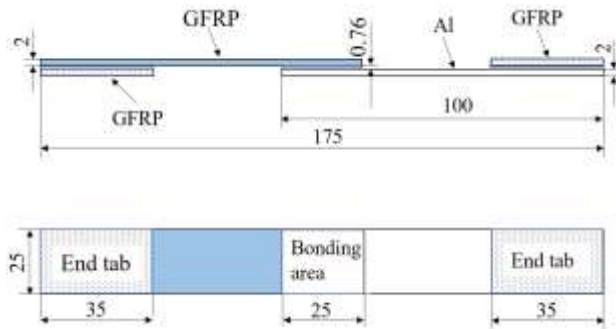


Figure 2. Dimensions of specimen for single lap-shear test according to ASTM D5868 (in mm)

### 3. Results and Discussions

#### 3.1. Contact Angle

The effect of atmospheric plasma treatments on the GFRP and aluminum surfaces was observed using water contact angle measurements. In Figure 3, it can be seen that the contact angle values of plasma-treated epoxy composite surfaces and aluminum decreased as compared to that of untreated surface samples. Moreover, when treatment time increased to 90 s, a longer air plasma treatment duration had more effect on the contact angle. Increasing plasma treatment time results in highly hydrophilic surfaces. The water contact angle of aluminum decreased from 90.1° for the untreated sample to 40.8° for the one treated with 50 kV-90s plasma. It is thought that the cleaning of the surface and the formation of hydrophilic polar groups on the surface with the effect of plasma treatment are the main reasons for the decrease in the contact angle [31]. Plasma treatment causes surface oxidation and the introduction of new polar functional groups into polymer surfaces, such as C=O, -OH, COOH, and C-O-C, which increases wettability [32].

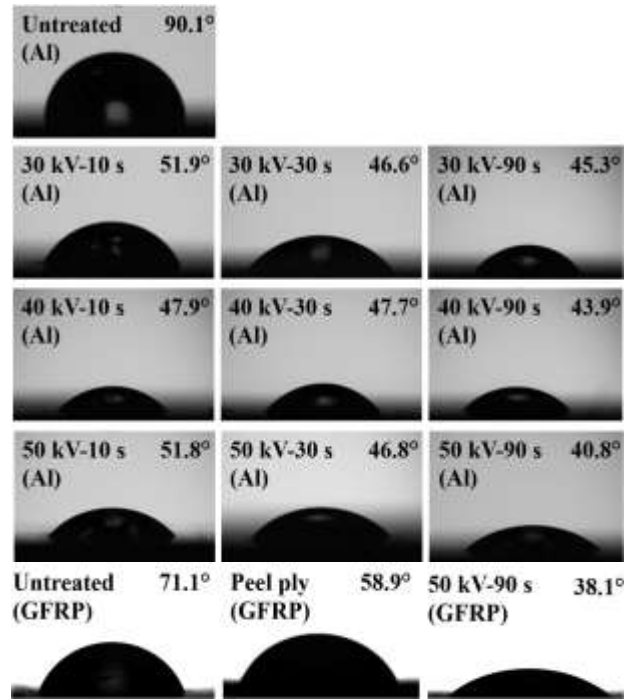
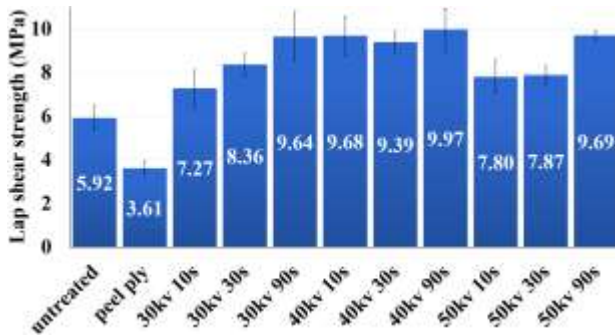


Figure 3. Measurements of water contact angle on Aluminum and GFRP substrates

#### 3.2. Lap shear strength

To evaluate the effect of atmospheric plasma treatments on the bonding strength, adhesive-bonded GFRP-Al joints were fabricated and lap-shear tests were carried out. Figure 4 shows variations in the lap-shear strength of adhesive-bonded GFRP-Al joints. The lap-shear strength of adhesive-bonded untreated GFRP-Al joints was only 5.92 MPa due to poor adhesion between GFRP and Al surfaces. This is due to the hydrophobic properties and low wettability of untreated GFRP surfaces. The maximum lap-shear strength of adhesive-bonded GFRP-Al joints fabricated from plasma-treated GFRP was 9.97 MPa. Active functional groups containing oxygen, such as the hydroxyl group (OH), carbonyl group (C=O), and carboxyl group (COOH), are formed on the composite surface. The active functional groups contribute hydrogen bonding with the adhesive, resulting in a strong adhesive force when compared to the Van der Waals force [33]. There is also good agreement with the results of water contact angle measurements, which show that wetting increases because of the increase in oxygen-related functional groups [32]. Surface adhesion may have improved, and bond strength increased due to the enhanced surface wettability of GFRP substrates resulting from the reduction in contact angle after plasma treatment. It has been observed that the shear strength values of all plasma-treated GFRP joints were higher than those of

untreated joints and joints treated with peel-ply surfaces. Rhee et al. reported that the peel strength was maximum when the water contact angle was minimum under optimal plasma conditions for the aluminum-CFRP adhesive joint [27]. The water contact angle decreased sharply after wiping an aluminum alloy with acetone and applying oxygen plasma surface treatment for 15 seconds in the study of Sperandio et al.. It has been noted that better flow and wettability of the adhesive results in excellent adhesive bond strength [34].



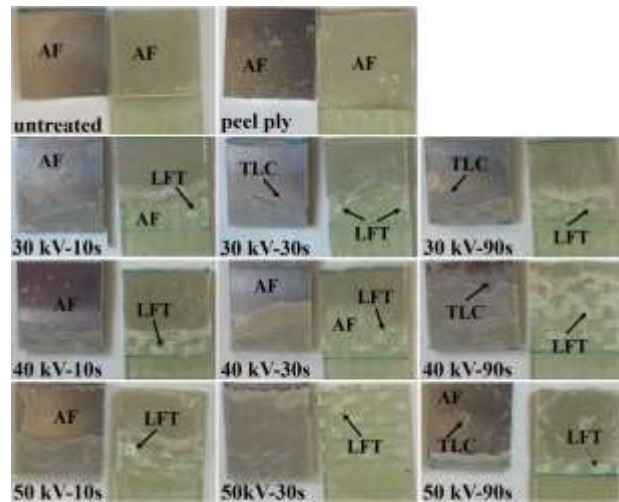
**Figure 4:** Lap shear strength values of adhesively bonded GFRP-Al joints with respect to untreated, peel ply and plasma parameters

### 3.3. Failure Modes

The surfaces of the fractured GFRP-Al joints were visually examined to identify the failure modes. Three different types of failure modes, known as adhesive, light fiber tear, and thin layer cohesive failure, were detected in the adhesively bonded joints as shown in Figure 5. Adhesive failure occurs when the adhesive is fully separated from one substrate, leaving only a small amount of adhesive residue on the surface of the other substrate. It can be seen that fractured surfaces of untreated and peel-ply surface treated substrates mainly exhibit adhesive failure mode. According to ASTM D5573, light-fiber-tear failure refers to failures that happen within the fiber-reinforced polymer, where thin layers of reinforcing fibers are visible on the surfaces. In addition, thin-layer cohesive failure is defined as occurring very close to the adhesive-adherent interface, characterized by a 'light dusting' of the adhesive on one of the substrate surfaces and a thick layer of adhesive remaining on the other substrate surface [35]. Light fiber tear failure modes were observed on all fracture surfaces of plasma surface-treated joints, regardless of plasma voltage and treatment time. Fibers were exposed in GFRP due to the separation of the epoxy on the top layer of the composite. It can be concluded that the adhesion of the adhesive to the surfaces is stronger than the adhesion between resin and fiber [36].

In addition to the light fiber tear failure mode, there was also a mixed (adhesive/thin layer cohesive/light fiber tear) failure mode in some fracture surface areas. Not only light fiber tear failure but also adhesive and thin layer cohesive mode occurred in plasma-treated joints. The lap shear strength of plasma-treated joints with mixed failure mode was higher than that of untreated and peel-ply treated joints with adhesive failure mode. In the study by Lee et al., mostly thin-layer cohesive and light fiber tear failure types were observed in the fractured samples of adhesively bonded GFRP joints. These failure types are closely related to peel failure, which predominantly occurs in adhesive-bonded joints due to peel stresses extending through the bond thickness direction [37].

As a result, adhesive failure mode occurred in untreated and peel-ply surface treated GFRP-Al joints with lower lap shear strength. Light fiber tear failure mode was dominantly observed on the fracture surfaces of adhesive joints produced by plasma surface treatment. In addition to the light fiber tear failure mode, mixed (LFT-TLC-Adhesive) failure mode was seen on the fracture surfaces of plasma surface-treated joints and had higher shear strength compared to the untreated and peel-ply treated joints.



**Figure 5.** Failure modes of adhesively bonded GFRP-Al joints with different surface treatments

### 4. Conclusions

To determine the relationship between wettability and adhesive bonding performance, air DBD plasma treatment was applied to aluminum alloy and GFRP substrates. This study experimentally explored how varying plasma voltages and treatment durations affect the mechanical performance of single lap adhesive joints. In addition, the effect of GFRP

substrate with peel ply surface treatment on the shear strength of the adhesive joint was examined. After lap shear testing, the fracture surfaces of the substrates were visually analyzed and the failure modes were determined.

- Air DBD plasma treatment increased the wettability of aluminum and GFRP substrates after modifying the surface with the appropriate plasma voltage and treatment time. According to water contact angle measurements, it was determined that the contact angle decreased regardless of the applied plasma parameters.
- Except for the applied plasma voltage of 40 kV, there is a good correlation between plasma treatment time and the lap-shear strength values. Although there were no significant differences between the mean shear strength values of adhesively bonded joints fabricated from plasma-treated GFRP and Al substrates, the highest shear strength was observed at 40 kV-90 s. An approximately 68% improvement in single-lap shear strength was obtained compared to the untreated joint. On the other hand, peel-ply surface treated joints had the lowest lap shear strength.
- The main failure mode for untreated and sanded bonded joints was adhesive failure. In the joints produced by plasma surface treatment, mixed (light fiber tear-adhesive-thin layer cohesive) failure mode occurred, and the main failure mode was the light fiber tear failure.

### Author Statements:

- **Ethical approval:** The conducted research is not related to either human or animal use.
- **Conflict of interest:** The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper
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