

Bonding dissimilar materials, specifically Glass Fiber Reinforced Polymer (GFRP) and Aluminum 6061 T651, at elevated temperatures, such as in fire accidents is challenging, where structural integrity and reliability are critical. We studied how surface roughness treatment affects the joint strength of three common adhesives in aircraft (Click Bond CB394-43, Loctite A9396, A9394) at 200 °C. The GFRP, composed of Gurit Prime 37 epoxy resin and E-Glass 7781 fibers produced through vacuum infusion with dimensions following ASTM D5868 standards, was lap shear tested with 2 mm/s stroke. Findings showed a substantial enhancement in joint strength due to surface treatment, sanding with 100-grid sandpaper for 20 seconds in parallel with the fiber direction, for all adhesives. A9396, A9394, and CB394-43 exhibited remarkable improvements of 1091.67 %, 45.92 %, and 30.09 %, respectively. The strain at break showed significant increases of 51.61 %, 121.95 %, and 100 %, respectively. Both surface-treated and untreated A9394 samples showed the highest strength among the adhesives. A9396 exhibited lower strength than CB394-43 without surface treatment, but it outperformed when surface-treated, highlighting its response to surface modification. Adhesive viscosity influences penetration on material surfaces, with A9396 being stiffer than the other adhesives. The analysis of ISO 4287 Ra values revealed that surface treatment led to increased roughness on the Aluminum surface while reducing roughness on the GFRP surface. These results offer valuable insights for optimizing GFRP-Aluminum bonding under elevated temperature conditions. Adjusting surface roughness significantly improves the interaction between Aluminum and GFRP with adhesives, resulting in enhanced joint strength. This knowledge can be applied in various engineering applications, particularly in industries where the performance and reliability of bonded joints are critical under high-temperature environments

Keywords: GFRP, Aluminum, surface treatment, joint, roughness, adhesive, high temperature, strength

UDC 691

DOI: 10.15587/1729-4061.2024.297904

IMPLEMENTING SURFACE TREATMENT AND ADHESIVE VARIATIONS FOR BONDED JOINTS BETWEEN COMPOSITE GFRP AND ALUMINUM AT 200 °C

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Received date 03.11.2023

Accepted date 29.01.2024

Published date 28.02.2024

How to Cite: Purnomo, H., Rifa'i, M. J., Purwoko, Vicarneltor, D. N., Ibadi, M., Setianto, M. H., Yudanto, M., Rizkyta, A. G. (2024).

Implementing surface treatment and adhesive variations for bonded joints between composite gfrp and aluminum at 200 °C.

Eastern-European Journal of Enterprise Technologies, 1 (1 (127)), 60–66. doi: <https://doi.org/10.15587/1729-4061.2024.297904>

1. Introduction

Aluminum and polymer are popular in the aerospace and automotive industries because of their excellent qualities such as strength, corrosion resistance, and lightness, particularly in the case of composites determining the effect of surface roughness treatment on adhesive joint strength [1–5]. Nevertheless, the optimal functioning of these two materials in specific applications often necessitates their interconnection. The association, as mentioned above, is commonly denoted as hybrid design and possesses the ability to generate a struc-

ture of higher quality [6]. There are three commonly employed methods of connection: mechanical connection, welding, and adhesive connection [7, 8]. To facilitate the installation of a rivet or bolt, mechanical connections necessitate the presence of a hole in the material. The process of welding necessitates the loosening of materials to establish a connection. Conventional mechanical connections (such as bolting, interference connections, and riveting) severely abrade the surface, seriously harm materials, and sometimes even permanently ruin fabric, making recycling impossible. A novel approach to connection that effectively shields fabric from harm

and circumvents the issues with stress concentration brought on by mechanical connections is bonding. On the other hand, the benefits of bonding are homogeneous stress distribution, a sizable load-bearing area, improved appearance, and increased stiffness [9, 10].

The process of adhesive connection necessitates using an adhesive substance that establishes a bond between two distinct materials. The adhesive connection method presents numerous advantages compared to alternative methods [11]. This approach effectively reduces potential damage that may occur during the drainage or heating process. Additionally, applying a glue coating aids in preventing corrosion, ensures a more uniform distribution of stress, and contributes to improved aesthetics.

Aircraft and shipbuilding frequently involve the use of aluminum and fiberglass for components such as wing structures and hulls. The lightweight and corrosion-resistant qualities of both materials, together with the strength of the connection, lead to high durability and fuel efficiency.

Aluminum Alloy 6061-T651 is the most commonly used for structural purposes. Because of its strong thermal conductivity and low density, aluminum heats up quickly when exposed to high temperatures. However, the reaction of AA6061-GFRP adhesive joint strength to heat propagation from fire damage raises concerns for practical applications. Furthermore, as the material heats, its strength rapidly decreases, making it sensitive to the impacts of fire and other high-temperature situations. Therefore, investigations assessing the effect of elevated temperature and surface roughness treatment on the adhesive joint strength of AA6061-GFRP under lap shear stress loading are scientifically relevant.

2. Literature review and problem statement

Adhesive joints for supersonic aircraft must withstand temperatures as low as (-55°C or colder) when traveling at high altitudes at subsonic speeds and as high as 200°C or higher at Mach 2 or higher [12]. Temperature also affects the adhesive joint due to the potential difference in thermal expansion of the material and the adhesive layer, as it causes extra stress in the bonded area when the temperature changes.

High temperatures in composite materials used in aerospace applications are those that exceed 200°C , as almost all matrices made of organic compounds begin to degrade and lose their usefulness at such temperatures [13]. In comparison, the ultra-high temperature category includes temperatures greater than $1,600^{\circ}\text{C}$ [14]. When aluminum and fiber composite joints in an aerospace structure are subjected to heat propagation, such as from fire, a host of challenges arises, potentially compromising the structural integrity of the aircraft. The diverse thermal properties of aluminum and fiber composites give rise to differential heat responses, resulting in stress concentrations at the joints. As heat spreads through the structure, the polymer matrix in fiber-reinforced composites undergoes degradation, leading to a loss of mechanical strength.

An adhesive is a polymer material; its mechanical properties are more temperature-sensitive. In their investigation into how temperature affected the mechanical characteristics of BFRP-Aluminum-alloy adhesive joints, other researchers discovered that the adhesive's tensile strength and Young's modulus dropped with rising temperatures [15].

In the previous study, the author limited it by adjusting the different types of sandpaper and sanding duration

to improve interface adhesion, so that the longer sanding time leads to an increase in aluminum surface energy, and increased surface energy results in improved mechanical adhesion, and demonstrated that the roughness of aluminum's surface has a significant influence on the strength of adhesion between aluminum and composite materials [16]. The most improved lap shear strength was obtained by using sandpaper P100, for 180 seconds, increased from 15.5 MPa to 20 MPa , about a 30 % increment. The study found that Aluminum with a surface roughness of $1.4\text{ }\mu\text{m}$ and a homogeneous surface is optimal for enhancing adhesive strength. However, this adhesion improvement observation is limited only to ambient temperature. In the instance of a fire in an airplane and ship, when heat propagates through the joint, there have been few observations on the effect of surface roughness on joint strength at high temperatures.

Surface roughness can be altered through various techniques, including laser shock processing, plastic media blasting, sandblasting, and ceramic shot peening. Other methods include metal shot peening with small steel particles and metal shock peening with large steel particles [17], leaching [18], and amplification. According to the study's findings, a combination of elements, including an ideal contact angle, Aluminum surface energy, optimal curing and theoretical maximum cohesive strength of epoxy glue, and appropriately managed surface roughness of the Aluminum alloy produced the highest lap shear strength. Surface roughness treatment influence on the adhesive bonding of metal and composite materials for high-performance structures was examined in the paper [19]. The surface of a metal or composite can be modified mechanically, chemically, or energetically to increase, in one way or another, the bonding strength and metal adhesion in the metal-composite connection. From the paper [16, 19], surface roughness treatment could improve metal-composite adhesive joint strength on mechanical testing at ambient temperature. Nevertheless, the study above has not been tested at high temperatures. This structure is very likely to be used at high temperatures.

Due to the difficulty of conducting adhesive joint strength characterization at high temperatures, the impact of surface roughness treatment on the strength of the aluminum-FRP adhesive joint at high temperatures has yet to be the subject of any particular research. However, aluminum-FRP structure sometimes must be applied at high temperatures. Therefore, aluminum-FRP adhesive joints with surface roughness treatment behavior between room temperature and 200°C need to be researched since it is important when applied to supersonic aircraft.

3. The aim and objectives of the study

This study aims to observe joining systems for GFRP composite and Aluminum at a temperature of 200°C by looking at the strength of three different adhesive systems. This research can be a recommendation for future GFRP-Aluminum adhesive joint applications at the specified temperature of 200°C .

To achieve this aim, the following objectives are accomplished:

- to observe the adhesive joint strength of three types of adhesive for GFRP-Aluminum Composite Joints at 200°C ;
- to determine the effect of surface roughness treatment on adhesive joint strength.

4. Materials and methods

4. 1. Object and hypothesis of the study

Our research focuses on the lap shear strength of the GFRP-Aluminum adhesive joint at 200 °C, which varies depending on surface roughness treatment and adhesive type. The primary hypothesis of the study is that surface roughness treatment will result in a surface that is more compatible with the adhesive and will increase lap shear strength at 200 °C. It is believed that the obtained lap shear strength data will be useful in developing GFRP and aluminum adhesive joints for use in the aerospace sector, as well as preventing structural failure of adhesive joints in the case of a fire.

4. 2. Materials

This experiment utilizes composite materials with E-Glass 7781 as reinforcement and 6061-T651 Aluminum Plate. Composites are produced using the Vacuum infusion method using a matrix Prime 37 epoxy resin from Gurit. There are three adhesives used, namely Epoxy Paste Adhesive Loctite EA 9394 [20] and Loctite EA 9396 [21] from Henkel Corporation Aerospace, and also Click Bond CB394-43 Epoxy Structural Adhesive from Click Bond Inc [22].

4. 3. Sample preparation

Destructive techniques for direct adhesion measurements involve quantifying the force necessary to fracture, tear, or separate surfaces at the interface [23]. This study used six samples as fiberglass-reinforced composite and aluminum adhesive joints, as seen in Table 1.

Table 1

Sample Adhesive Joint GFRP/Al

Sample	Adhesive	Surface Treatment
EA9396	Loctite EA 9396	No
EA9396-SP	Loctite EA 9396	Yes
EA9394	Loctite EA 9394	No
EA9394-SP	Loctite EA 9394	Yes
FAC	Click Bond CB394-43	No
FAC-SP	Click Bond CB394-43	Yes

The sample codification was based on the adhesive used and whether surface treatment was done. The number 9396 indicates that the adhesive used is Loctite EA 9396. Then, the sample that used Loctite EA 9394 as the adhesive was marked by 9394 in the sample code. On the other hand, the sample code for the Click Bond CB394-43 adhesive that was used as adhesive had a «C» on it. The «SP» code was used to sign that surface treatment was done on the sample.

4. 4. Surface treatment procedure

A composite material consisting of Glass fiber Reinforced Polymer (GFRP) alloy, precisely Aluminum type 6061 T651, was utilized for this study. The material was precisely cut to a length of 100 mm, a width of 25 mm, and a thickness of 3 mm. The sander belt tool is used to apply pressure to the ends of a 2x2 cm surface area with a 100-grid for 20 seconds in parallel with the fiber direction. This sample configuration follows the ASTM D5868 Lap Shear Adhesion Test for Fiber Reinforced Plastics (FRP). The subsequent step involves using a clean cloth to cleanse the finished surface. Glass-fiber rein-

forced polymer (GFRP) and Aluminum composite surfaces are suitable for adhesive application. The Aluminum and composite materials were joined together using the adhesive application by applying adhesive one-sided on a GFRP surface with a wet thickness of 1 mm and then, with Aluminum, secured with a clamp fixture with 1 Kg pressure for 5 days at ambient temperature for the curing process to occur [20–22], following the adhesives data sheets, which recommend 3 to 5 days for curing. This bonding procedure produced an adhesive layer that was 0.04–0.05 mm thick when dry.

The research subject was a single solid lap adhesive joint of selected structural materials. Three types of epoxy adhesives were utilized to make adhesive joints: Click Bond CB394-43 Epoxy Structural Adhesive from Click Bond Inc. [22], Epoxy Paste Adhesive Loctite EA 9394 [20], and Loctite EA 9396 [21] from Henkel Corporation Aerospace. It is appropriate for the analyzed adherents and cures quickly at room temperature. All of the glues used in this research are typical because they are composed of epoxy-type polymers. Moreover, they are suggested to be used at high temperatures so the glues that are used in this experiment are comparable [20–22].

The surface sample's preparation or treatment dramatically impacts how much weight the aluminum-polymer adhesive joints can support. Using abrasive paper, the most practical material for mechanically treating adherents, the sample preparation process included an abrasive mechanical treatment step. The rationale behind the selection of this strategy is its high efficiency, accessibility, affordability, and ease of usage under a variety of scenarios. Another significant advantage is that this method requires little effort to ensure that the machined surface displays marks in all directions. P100 abrasive paper was used for mechanical treatment during experimental testing.

4. 5. Roughness test

An object's different parts have many different shapes and sizes, encompassing both structural characteristics and outcomes resulting from the production process. The parameter denoting rigidity is quantified regarding the Roughness Average (Ra). The rigidity parameter known as Ra is extensively utilized globally [24].

Roughness measurements are conducted on aluminum and composite surfaces before and after surface treatment. The measurement of surface roughness, specifically the Ra value, was conducted using the Roughness And Contour Tester Kosaka Lab. SEF800-G. The measurement parameters included a cutoff of 0.8 mm, a speed of 0.2 mm/s, and a measurement length of 1 cm. The measurement procedure followed the guidelines outlined in ISO 4287 [25].

4. 6. Lap shear testing using a universal testing machine (UTM)

The Universal Testing Machine (UTM) Shimadzu AG-X plus, with a capacity of 50 kN, was used to determine the lap shear strength of the adhesive from various GFRP/Al adhesive joints. Compact Systems Thermostatic Chamber TCE-N300A also gives high-temperature conditions during lap shear tests. Before the pull test, the specimen was well attached to the Jig machine, and the chamber was installed on the sample area to be heated to a temperature of 200 °C. Once the temperature reaches 200 °C, the chamber opens and is ready for the pull test. Samples in the one-on-one and pull test using a speed of 2 mm/s. Testing has been conducted in the Testing Laboratory of the Polymer Technology Center.

5. Results of lap shear test at 200 °C and roughness test of GFRP-aluminum composite adhesive joints

5. 1. Lap shear properties of GFRP-aluminum adhesive joints at 200 °C

The lap shear test results are shown in Table 2. Applying surface treatment resulted in a significant enhancement of the lap shear strength across all experimental conditions. The strength of Loctite A9396, Loctite A9394, and Click Bond CB394-43 increased by 1091.67 %, 45.92 %, and 30.09 %, respectively. The utilization of Loctite A9394 has been found to yield the most substantial adhesive joint strength. It should be noted, however, that the increase in adhesive joint strength due to the application of the surface treatment of Loctite, A9396, is primarily determined by the surface treatment applied.

Table 2

Lap Shear test results

Sample	Lap Shear Strength (MPa)	Strain at Break (%)
FA9396	0.12±0.04	0.31±0.09
FA9396-SP	1.43±0.16	0.47±0.07
FA9394	1.35±0.26	0.41±0.19
FA9394-SP	1.97±0.12	0.91±0.23
FAC	1.03±0.10	0.35±0.05
FAC-SP	1.34±0.28	0.70±0.50

The specimens subjected to surface treatment exhibited increased strain at the fracture point. The strain at break increased by 51.61 %, 121.95 %, and 100 % for Loctite A9396, Loctite A9394, and Click Bond CB394-43. The sample with Loctite A9394 had the highest strain at break value. The strain at break data suggests that the surface treatment process had a beneficial effect on the strain at break.

Fig. 1, 2 depict surface observations of the test object after testing. The adhesives left on the adhesive joint's surface from Fig. 1, 2 show different behavior with various treatments and adhesive types.

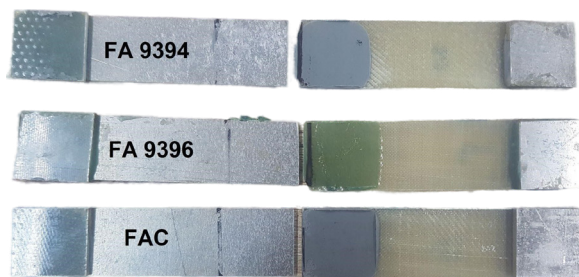


Fig. 1. Test specimens without treatment

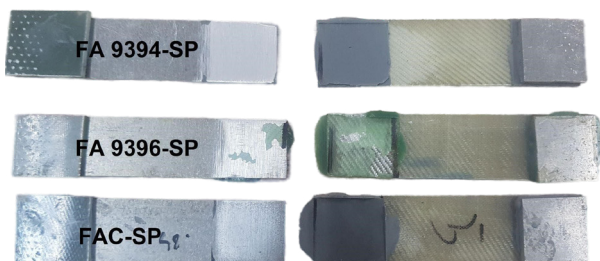


Fig. 2. Test specimens with treatment

All adhesives were left only on composite surfaces for the samples without surface treatment. When surface treatments were done, Loctite A9394 and Click Bond CB394-43 adhesives were left only on composite surfaces. On the other hand, Loctite A9396 adhesive was left partially on the Aluminum surface, and most of the adhesive was on the composite surface when surface treatments were applied.

5. 2. Surface roughness of GFRP and aluminum

The roughness test is conducted to measure the unevenness of the surface of an object or substrate. This unevenness can be measured using roughness parameters, such as Ra (arithmetic mean) [26]. The study's objective is to perform an analysis of the rigidity of Aluminum and composite to examine the alterations in rigidity parameters resulting from surface roughness treatment, with the untreated surface serving as a baseline for comparison. Table 3 shows the results of the test for rigidity.

Table 3

Comparison roughness test results

Specimen Material	Without Surface Treatment	Surface Treatment
Alumunium	0.24 μm	1.96 μm
Composite	13.6 μm	2.42 μm

Surfaces with varying degrees of roughness can exhibit tiny characteristics that may differ in size. The presence of irregularities can have an impact on the interaction between the surface and the adhesive that is put on it [27]. The roughness test results of aluminum after surface treatment are shown in Fig. 3. On the other hand, Fig. 4 represents the composite roughness test result of composite material that has been surface-treated.

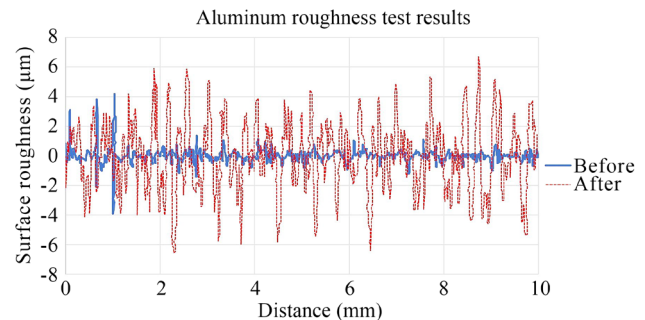


Fig. 3. Aluminum roughness test results

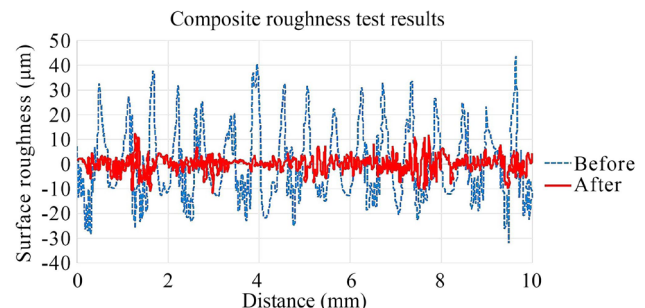


Fig. 4. Composite roughness test results

The roughness of Aluminum is due to the surface treatment of Aluminum from 0.24 μm to 1.96 μm, as shown in Fig. 3;

increasing surface roughness positively affects the surface contact area between adhesive and substrate, resulting in increased adhesion and bond strength [28]. However, in the case of the composite material, surface treatment results in a reduction in roughness value from 13.6 μm to 2.42 μm , as observed in Fig. 4.

6. Discussion of roughness test and lap shear properties results at 200 °C of surface-treated GFRP-aluminum adhesive joint with several types of adhesive

The lap shear test results from Table 2 show that adhesive joint strength with Loctite A9396, Loctite A9394, and Click Bond CB394-43 increased by 1091.67 %, 45.92 %, and 30.09 % at 200 °C, respectively. This indicated that, in general, the surface treatment with GFRP and Aluminum increased lap shear strength at 200 °C. Surface treatment with grid 100 sandpaper for 20 seconds can modify Aluminum and composite surface roughness, increasing by 716.67 % and decreasing by 82.21 %, respectively, as seen in Table 3, Fig. 3, 4. Interestingly, despite a drop in roughness for Aluminum, the strength of the adhesive joint continues to increase, as evident from the data presented in Table 2. This phenomenon can occur in some instances, such as when adhesion is established on irregular or uneven surfaces. In such scenarios, the adhesive strength may be diminished due to increased gaps or void spaces between the adhesive surface and the substrate [29]. This observation suggests that there exists an ideal surface roughness value that yields the highest adhesive joint performance. Based on the findings of the paper [30], it has been determined that the ideal surface roughness influences the tensile strength of adhesion. The ideal value falls within the range of 3 to 6 micrometers. According to the paper [31], Aluminum adhesive joints found that the value of surface roughness necessary to achieve the optimal adhesive strength decreases within the range of 1.75–2.5 μm .

The surface roughness of Aluminum materials can significantly impact the quality of the coatings applied [32]. The smoothness and uniformity of the surface are essential for the successful attachment of the adhesive to the composite substrate. Insufficient surface roughness may result in sub-optimal wetting and a diminished contact area between the adhesive and substrate, weakening bonds and compromising coating quality. There is a direct relationship between the surface's roughness and the coating's quality. The rougher the surface, the more tightly the adhesive adheres to the surface imperfections, leading to a more vital interlocking force and improved bonding performance. Better interaction is gained when the surface roughness value is close to the optimum roughness value. The interaction of adhesive and Aluminum plays a more significant role in the adhesive joint strength of GFRP/Al adhesive joints. Surface treatments, including abrasion, viscoelastic magnetic abrasion [33], and chemical etching [34], as well as surface activation, can modify the properties of the material surface, thereby impacting the adhesive's capacity to wet the surface and establish a robust bond effectively.

From Table 2, Loctite A9394 achieved the highest strength value for both samples with surface treatment and without treatment. It indicates that Loctite A9394 has the highest mechanical properties at 200 °C and better interface on the Aluminum surface. Loctite A9396 adhesive joint strength value is lower than Click Bond CB394-43 for samples with-

out surface treatment, but the value is higher for samples with surface treatment. It indicates that Loctite A9396 has a better strength but poor interaction with untreated Aluminum. However, after surface treatment, Loctite A9396 has a better interface. It is assumed that Loctite A9396 has a lower viscosity than Click Bond CB394-43 due to the lower content of filler [21], so it has better penetration on treated surfaces. From Fig. 1, 2, findings indicate that the adhesives exhibit detachment from the Aluminum surface while remaining adhered to the GFRP composite surface. This finding demonstrates that adhesives are more compatible with composite surfaces than Aluminum. Additionally, it suggests that the interaction between Aluminum and the adhesive significantly influences the strength of the junction. Adding fillers to an adhesive binder results in the insolubility of the adhesive. This is due to the solid nature of the fillers and their relative non-adhesion. Additionally, adding fillers can alter the rheological and other characteristics of the adhesive [35]. The mechanical strength and viscosity of the adhesive impact the adhesive joint strength of the GFRP/Al adhesive joints. The lower the viscosity, the better the penetration on the treated surface.

There are some limitations of this research that can be considered before application. First, the limitation of this research that should be considered is the temperature when the adhesive joint was applied. In this research, the temperature of the test was only at 200 °C. Then, the surface treatment process of this research is also limited because it only uses P100 abrasive paper. Third, the manufacturing process of composite plates that used peel ply should be considered when designing the adhesive joint. Last, the adhesive type used was only Loctite EA 9394, Loctite EA 9396, and Click Bond CB394-43.

Several study shortcomings can be identified, and techniques can be proposed for overcoming them in future research. One of these disadvantages is that the observation had been done at a specific temperature. The behavior of the bonded materials may differ at temperatures above or below this range, and the findings may not be directly extrapolated to different temperature conditions. Future research efforts could conduct testing at several temperatures to produce a temperature-dependent bonded material profile to address this issue effectively. This can aid in identifying any crucial temperature thresholds at which the material's behavior changes significantly. It should also consider testing at intervals within the temperature range that the bonded materials are anticipated to undergo in real-world applications. The other disadvantage is that an optimum surface roughness has yet to be achieved. This research suggests an ideal surface roughness value for optimal adhesive strength is 3 to 6 micrometers based on reference. However, this ideal range might be specific to the materials and conditions studied and may not be universally applicable to all adhesive bonding scenarios. Further research into surface roughness optimization can be conducted to acquire the best bonding strength by considering surface treatment methods, pressure, and time.

7. Conclusions

1. After surface treatment with sandpaper, adhesive joint strength with Loctite A9396, Loctite A9394, and Click Bond CB394-43 increased by 1091.67 %, 45.92 %, and 30.09 %

at 200 °C, respectively. After surface treatment, the strain at break at 200 °C increased by 51.61 %, 121.95 %, and 100 % for the respective adhesive materials, Loctite A9396, Loctite A9394, and Click Bond CB394-43. This is because the surface treatment process can change the roughness of the material's surface, affecting the adhesive's ability to wet the surface and form a strong bond effectively.

2. Loctite A9394 has the best mechanical properties at 200 °C, and adhesive joint lap shear strength test results are influenced by surface treatment roughness and adhesive type. This is due to the low filler content and viscosity of Loctite A9394. The mechanical strength and viscosity of the adhesive influence the adhesive joint strength of the GFRP/Aluminum adhesive joints. The better the penetration on the treated surface, the lower the viscosity.

Conflict of interest

The authors declare that they have no conflict of interest about this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

Financing

This research has been generously funded by the National Research and Innovation Agency through the Rumanah Program Research Organization for Aeronautics and Space 2024 (2/III.1/HK/2024).

Data availability

The manuscript has associated data in a data repository.

Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies when creating the current work.

Acknowledgments

The study was conducted in the laboratory of the National Research and Innovation Agency (BRIN).

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