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Synergistic effect of surface treatment and adhesive type on bonding performance of thin Al6061 joints for automotive applications

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ABSTRACT

The effects of different surface treatments (grinding, plasma surface treatment, and grinding followed by the plasma (GrPl) process) and various adhesives (cyanoacrylate, Bisphenol A (BPA)-based, and Bisphenol A and Bisphenol F (BPA/F)-based adhesive) on the bonding performance of single-lap joints manufactured from thin Al6061-T4 aluminium alloy sheets were investigated in this study. Contact angle measurement was carried out to evaluate the influence of surface treatments on the wettability of the surface of the aluminium alloy adherends. The contact angle on the surface of the untreated sheet was measured to be 71.8°. However, after applying the GrPl process on the ground surface, the contact angle could be reduced to 47° and 25.9° in the parallel and perpendicular directions to the grinding direction, respectively. Shear testing was performed to evaluate the mechanical behaviour of the single-lap joints of aluminium alloy sheets. The shear strengths of BPA/F-based, cyanoacrylate, and BPA-based adhesive-bonded joints produced from ground adherends improved by 47 %, 54 %, and 72 %, respectively. The synergistic impact of GrPl treatment resulted in 126 %, 123 %, and 174 % enhancement in shear strength of BPA/F-based, cyanoacrylate, and BPA-based adhesive-bonded joints, respectively. When the temperature was raised from room temperature to 60 °C, the bonding performances of all single-lap joints deteriorated due to the decrease in the strength of the adhesive. However, the joints treated with plasma had the highest shear strength at 60 °C. While the BPA-based adhesive had the most beneficial impact on improving the shear strength of single-lap joints at room temperature, the opposite result was obtained at 60 °C. All the findings have indicated that the influence of adhesive on the bonding performance of aluminium alloy joints varied significantly depending on the surface treatment and temperature.

1. Introduction

Adhesive bonding is a popular method for joining components in the automotive and aerospace industries because it offers several advantages over conventional methods, such as improved mechanical properties and simplicity in production [1–3]. In many applications, the lightweight of the parts to be bonded is crucial, and aluminium alloys are commonly employed to meet this demand [4]. The components produced from aluminium alloy sheets are joined using various methods such as welding and riveting [5–7] and this is a very critical issue for thin sheets. Some of these methods have a negative influence on the mechanical properties of the joints. For instance, when joining the sheets by riveting, the stress concentration around the rivet hole may cause the properties of the joints to be weak; additionally, the rivet head increases the weight of the joint [8]. The lap-shear strength of adhesively bonded and hybrid riveted-bonded aluminium alloy joints was compared in the study of Beber et al. [9]. They revealed that adhesive-bonded joints exhibited a higher lightweight potential (lap-shear strength divided by joint weight) compared to riveted-bonded joints. Domitner et al. [10] investigated the static and fatigue strength of self-piercing riveted, adhesive bonded, and hybrid (adhesive + riveting)-bonded 6061 aluminium alloy joints, and they suggested that the adhesive layer provided the main contribution to the mechanical performance of joints, whereas the rivets contributed only minimally. In bonding the sheets by welding method, the excessive heat generated during the process, causes negative impact on the microstructure and mechanical properties of the joint [11]. These drawbacks that arise in these methods do not occur in adhesive bonding. Furthermore, compared to welding and riveting, adhesive bonding provides a continuous bond, resulting in more uniform stress distribution and improved

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Nomenclatures

AF	Adhesive Failure
BPA	Bisphenol A
BPA/F	Bisphenol A and Bisphenol F
CF	Cohesive Failure
DBD	Dielectric Barrier Discharge
F	Load
GrPl	Grinding followed by the plasma
HV	High Voltage
l	Overlap length
R_a	Arithmetical mean roughness
R_z	Mean roughness depth
τ	Shear strength
w	Overlap width
XPS	X-ray Photoelectron Spectroscopy

mechanical properties of the adhesive-bonded joints. Antelo et al. [5] investigated the performance of welding and adhesive bonding of structural components. They suggested that the static strength of adhesive bonding was higher than the welded joints.

The performance of adhesive bonding is strongly dependent on factors such as adhesive type and surface treatment type [12,13]. Epoxy-based adhesives are widely utilized for bonding aluminium parts in various applications, including automotive and aerospace industries [14–19]. They are primarily monomeric or oligomeric compounds that bond to substrates [20]. Araldite is a two-component epoxy-based high-strength adhesive. Loctite is a one-component cyanoacrylate-based (Loctite 401, etc.) or two-component epoxy-based (Loctite EA 9658, etc.), fast-curing adhesive used in industrial applications. Carvalho and Campilho [21] investigated the mechanical behaviour of single-lap 6082 aluminium alloy joints produced using various epoxy-based adhesives. Araldite adhesive exhibited good performance in terms of the mechanical behaviour of the aluminium joints. Safari et al. [12] studied the effect of adhesive type on the shear strength of 2024 aluminium alloy joints. They suggested that the lap shear strength of high-viscosity epoxy-based (Araldite 2015)-bonded joints was higher than that of a low-viscosity epoxy-based adhesive-bonded joints. Although epoxy-based adhesive is a strong adhesive used in aluminium joints, as seen in the studies, it is still being developed to enhance the mechanical performance of the adhesive-bonded aluminium joints. Rachid et al. [22] studied the effect of adding silica nanoparticles to reinforce epoxy adhesive on the shear strength of single-lap joints of 2024 aluminium alloy. They suggested that the inclusion of nano-silica had a noteworthy influence on enhancing the mechanical performance of the joints. It should be noted that the properties of adhesives can change under certain conditions, such as high temperature and long-term loading, which can have a negative impact on the mechanical properties of adhesive-bonded joints. Ghasemvand et al. [23] examined the strength and creep behaviour of Araldite 2011-bonded single-lap 6060 aluminium alloy joints at different temperatures: 23 °C (room temperature (RT)) 45 °C, and 55 °C. It has been suggested that the mechanical performance of the joints decreased as the temperature increased, attributing this to the loss of stiffness in the polymer chains of the adhesive and the increased softening of the adhesive. Hirulkar et al. [24] studied the strength of single-lap joints of 6082 aluminium alloy bonded with Araldite 2015 and Araldite AV138 adhesives, subjected to hygrothermal aging along with cyclic thermal shocks at temperatures of −5 °C, room temperature, and 50 °C. It has been reported that the thermal shock significantly reduced the mechanical strength of the joints, and it was observed that the Araldite 2015 adhesive-bonded joint had a higher failure load compared to the Araldite AV138 adhesive-bonded joint.

Another important factor that affects the mechanical properties of adhesive bonding is the surface preparation and modification of the components. Surface treatment processes contribute to improved bond strength; this is due to the following factors: (i) increased surface bonding area, (ii) mechanical interlocking between adhesive and adherend, and (iii) enhanced wettability resulted from modifications (plasma activating, etc.) of surface chemistry. Among surface treatment processes, mechanical treatments such as grinding and sandblasting not only remove contaminants such as lubricant but also roughen the surface to increase the bonding area, which improves the mechanical properties of adhesive-bonded joints [25,26]. Liu et al. [25] investigated the influence of surface roughness on the lap shearing strength of one-component epoxy-bonded aluminium joints. The lap shear strength of adhesive-bonded joints was reported to improve as surface roughness increased due to a larger surface area and improved mechanical interlocking. Excessive roughening, on the other hand, reduced the wettability of the surface of aluminium alloys, resulting in a reduction in the lap shear strength of joints. A similar trend was observed in the study of Ghumatkar et al. [27], in which they investigated the effect of different adherend surface roughness on the adhesive bond strength of two-component epoxy-based adhesive-bonded 6063 aluminium alloy single strap joints and found that shear strength was higher in grinded specimens with higher surface roughness than in untreated specimens, but then decreased as surface roughness increased. Sandblasting is another method for increasing the surface roughness of an adhesively bonded joint to improve its performance. Li et al. [26] studied the impact of sandblasting variables (sandblasting pressure and abrasive particle size) on the epoxy-bonded Al–Li alloy sheets. The surface roughness increased with increasing abrasive size and pressure, which had a favourable effect on the wettability and bonding capabilities of the surface of Al–Li alloy sheets. However, excessive roughness may hinder the efficient penetration of the adhesive into the surface grooves, resulting in a reduction in shear strength [28]. Alderucci et al. [29] formed grooves in various patterns on the surface of 5083 aluminium alloy, and they proposed that, unlike traditional mechanical processing, which can only change roughness with limited useful impacts, the formation of grooves created a controlled surface profile by promoting the anchoring effect of the adhesive, resulting in the strength of the adhesively-bonded single-lap aluminium alloy joints. Some researchers investigated the influence of chemical treatments on the surface roughness and adhesive bonding performance [15,30,31]. Saleema et al. [30] applied a sodium hydroxide solution to the surface of 6061 aluminium alloy at various treatment times. Surface roughness increased as treatment time increased. The shear strength of the epoxy-bonded 6061 aluminium alloy joint improved as the surface roughness increased. Despite providing the highest surface roughness, the longest treatment duration did not give a further increase in shear strength. Ozun et al. [31] investigated the effects of both chemical (etching with a solution of sulphuric acid and sodium dichromate) and mechanical (abrading) surface pretreatments on the adhesion strength of the epoxy-bonded 7075 aluminium alloy joints. The etched surfaces were reported to have higher surface roughness than the abraded ones, and it was suggested that the improvement in shear strength of the single-lap aluminium alloy joints was due to the significant increase in roughness.

Surface modification procedures such as plasma treatment, laser surface preparation, and corona treatment have become preferred methods to improve the performance of adhesive bonding [32–40]. Because of their great efficiency, plasma treatment has become more prominent compared to other surface treatment methods in recent years, particularly for aluminium alloys [39,41]. Wang et al. [42] studied the effect of atmospheric pressure plasma treatment on the strength of a one-component epoxy-bonded 5052 aluminium sheet. They revealed that plasma treatment reduced the contact angle of the liquid on the surface of aluminium alloy, improving the wettability and strength of an epoxy-bonded 5052 aluminium joint. Saleema et al. [34] applied the atmospheric pressure plasma oxidation process onto the surface of 6061 aluminium alloy sheet. It has been proposed that the good bonding between the hydroxyl groups on the surface the aluminium alloy and molecules in epoxy resin and the surfaces exposed to oxygen plasma could result in enhanced adhesion strength as well as improved surface wettability. Ba et al. [43] investigated the impact of atmospheric pressure low-temperature plasma on the surface bonding performance of epoxy-bonded aluminium alloy single-lap joints. They reported that the shear strength of the joints increased due to a decrease in contact angle and an increase in wettability. They also suggested that the amount of oxygen-containing functional groups on the surface of the aluminium alloy increased, improving wettability and leading to the formation of chemical bonding.

The outcomes of these studies demonstrate that atmospheric plasma surface treatment, as an innovative surface treatment, can significantly enhance the mechanical performance of adhesive-bonded aluminium alloy joints. Additionally, grinding, as a mechanical surface treatment, has also been observed to have a positive impact on the mechanical properties of these joints. These findings suggest that implementing a combination of two distinct surface treatments would further enhance the mechanical properties of adhesive-bonded aluminium alloy joints. However, it is important to note that there is a limited number of studies that have examined the combined effects of surface treatments on the mechanical properties of these joints. Furthermore, it should be acknowledged that only one type of (epoxy-based) adhesive was utilized in these studies. The present work aimed to assess not only the synergistic impacts of various surface treatments (grinding, plasma, and grinding + plasma) but also the influence of different adhesive types (cyanoacrylate, epoxy (BPA and BPA/F)-based) on the shear strength of adhesive-bonded single-lap Al6061-T4 joints. The second aim of the study was to conduct a comprehensive investigation on thinner (1 mm) aluminium alloy sheets than those examined in previous literature studies on this subject. The third objective was to investigate the mechanical performance of Al6061-T4 joints not only at room temperature but also at an elevated (60 °C) temperature. In addition to the important application areas of Al6061-T4 sheets in various sectors, its easy formability and high ductility give the high potential of widening its usage not only in the automotive and aerospace industries but also in battery housing applications in the near future.

2. Materials and methods

2.1. Materials

A commercial T4 temper 6061 aluminium alloy (Al6061-T4) sheet with a thickness of 1 mm was used to manufacture the single-lap joints. A thin Al6061-T4 sheet was preferred because it is widely employed in automotive and aerospace applications due to its lightweight, high resistance to corrosion, and good mechanical properties [41]; for example, it is a commonly used material in components such as battery cover plate [44]. The chemical composition and the mechanical properties of the Al6061-T4 used in this study were given in Tables 1 and 2.

To bond the adherends in the production of the single-lap joints, three different adhesives were used: a one-component cyanoacrylate adhesive (Loctite® 401, Henkel Adhesives, Dublin, Ireland), a two-component Bisphenol A (BPA)-based adhesive (Polires® 114 for resin and Epilox® M 1171 for hardener, Polikem, Istanbul, Turkey), and a two-component Bisphenol A and Bisphenol F-(BPA/F)-based adhesive (Araldite® 2011, Huntsman Advanced Materials, Basel, Switzerland). The properties of adhesives given in Table 3 were taken from the data sheets provided by the manufacturers. Both cyanoacrylate and BPA/F-based adhesives were preferred for their ability to cure rapidly and provide high strength, and the BPA-based adhesive was chosen because of its low viscosity.

Table 1
Chemical composition of Al6061-T4 alloy.

Elements	Mg	Si	Fe	Cu	Cr	Zn	Ti	Mn	Al
(wt.%)	0.8–1.2	0.4–0.8	≤0.7	0.15–0.4	0.04–0.35	≤0.25	≤0.15	≤0.15	Bal.

Table 2
Mechanical properties of Al6061-T4 alloy.

Tensile strength (MPa)	Yield strength (MPa)	Elongation (%)	Shear strength (MPa)	Shear modulus (GPa)
241	145	22	165	26

2.2. Preparation of adherends

2.2.1. Cutting Al6061 sheets

Fig. 1 shows the stages of the manufacturing process for adhesive-bonded single-lap joints. Firstly, Al6061-T4 sheets with a thickness of 1 mm were cut into dimensions of 25.4 mm in width and 101.6 mm in length to manufacture single-lap joint specimens in accordance with ASTM D1002. All Al6061-T4 adherends were cleaned in an ultrasonic bath to remove surface contamination. The cleaning process was carried out in an ultrasonic bath for 5 min at a frequency of 37 kHz in a solution of 10 vol.% concentrated ethanol and pure water. The cleaned sheets were subsequently dried in a desiccator cabinet at a temperature of 25 °C.

2.2.2. Application of surface treatments onto Al6061 sheets

Following the cleaning process, surface treatments were applied to the surface of the adherends in order to improve the adhesion of the adhesive to the surface of the adherends. The surface treatment methods used were grinding, plasma treatment, and a combination of grinding followed by plasma treatment (GrPl). Grinding was performed to the surface of the adherends to enhance the contact surface and mechanical interlocking between the surface of the adherend and the adhesive in order to improve adhesion and mechanical properties [45]. In this study, a special tool was used in the grinding process to fix the specimen and to keep the load applied for grinding the surface of the specimen constant. The grinding process was performed on the surface of the adherends in a single direction that was perpendicular to the loading direction of the adhesive joints for shear testing using three different sandpapers with mesh numbers of P180, P320, and P600. The grinding process was carried out for a duration of 10 min.

Atmospheric plasma treatment can be an alternative and innovative method to chemical and mechanical surface treatments such as etching and grinding. This process is an efficient, environmentally friendly, and cost-effective method of cleaning (removing the contaminants) and activating chemical bonds on the surface of the adherend in order to improve adhesion bonding; furthermore, the atmospheric plasma treatment contributes to improving the bonding strength between the surface and the adhesive [42,46–49]. The atmospheric pressure plasma method is conducted using different gases such as argon, nitrogen, and air. It has been suggested in the literature that air plasma is more beneficial than the others in increasing aluminium wettability and improving the lap-shear strength of adhesive-bonded aluminium joints [42]. For these reasons, atmospheric plasma surface treatment was performed on the surface of the aluminium alloy adherends using a PTP 22-01 model DBD air plasma system (PACEM Technology, Izmir, Turkey), as shown in Fig. 2. A dielectric barrier discharge (DBD) electrode was used to generate active air plasma. The DBD electrode was

Table 3
Properties of adhesives used to produce single-lap joints made from Al6061-T4 sheets.

Property	Adhesive type		
	Cyanoacrylate	BPA-based	BPA/F-based
	One component	Two-component	Two-component
Chemical compound	Cyanoacrylate	Diluted Bisphenol A epoxy resin with modified polyamine adduct hardener	Bisphenol A and Bisphenol F epoxy resin with N(3-dimethylaminopropyl)-1,3-propylenediamine hardener
Viscosity at 25 °C (mPa s)	100–120	600-1200 for resin, 160–260 for hardener	30.000–45.000 for resin, 35.000 for hardener
Mixing ratio	–	2:1	1:1

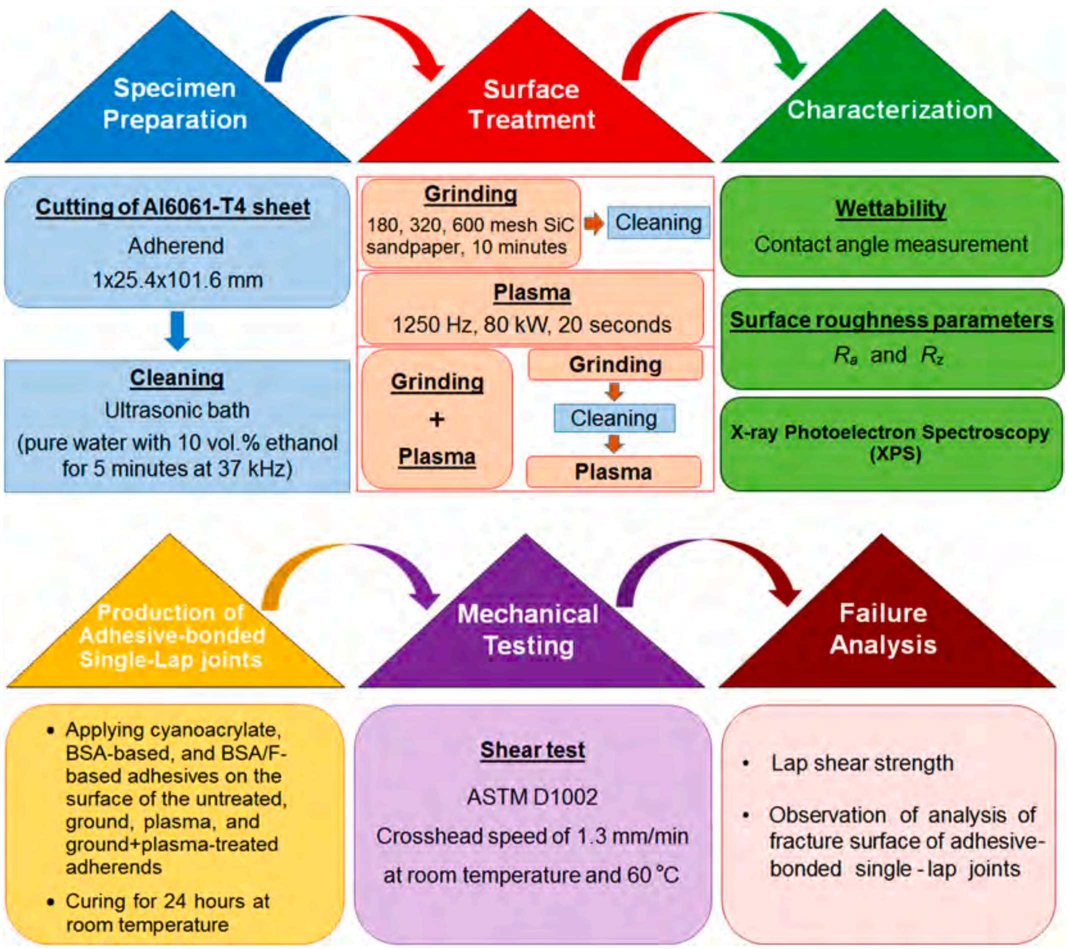

Fig. 1. Schematic diagram of manufacturing stages, characterization, and experimental studies for adhesive-bonded single-lap joints.

constructed by covering copper plates (10 mm thick) with a 1 mm thick glass. The remaining part of the copper plate was then enclosed in a polyethylene housing to provide insulation for the exposed surfaces. In the literature, the process parameters used in the studies investigating the effects of atmospheric pressure plasma treatment on the mechanical properties of adhesive-bonded joints are different from each other and have different values in a wide range. The suitable process parameters in the current investigation were determined by assessing these studies [34,46,49–52]. Plasma treatment was performed for 20 s using a frequency of 1250 Hz and a power of 80 W. The discharge gap was fixed as 1 mm.

Untreated, ground, plasma-treated, and GrPl-treated aluminium alloy adherends were categorized into eight groups, as shown in Table 4. Five adherends were prepared for each group for the measurement of surface roughness and contact angle.

2.3. Characterization

2.3.1. Surface roughness

The surface roughness is a critical factor in increasing the shear strength of single-lap adhesive joints since it impacts the contact angle and the effective bonding area of the adherend. As the surface of the adherend is roughened, the bonding (contact) area and wettability of the adhesive on it increase, promoting a good mechanical interlock effect between the adhesive and the adherend. In the studies conducted in the literature to investigate the effects of surface treatments on the properties of adhesively bonded single-lap aluminium joints, surface roughness parameters: R_a and R_z, are commonly used to assess the quality of the surface of the aluminium alloy adherends [53–57]. In the present study, these two surface roughness parameters were measured to determine the surface roughness of untreated, ground, plasma-treated, and GrPl-treated surfaces of the specimens. R_a

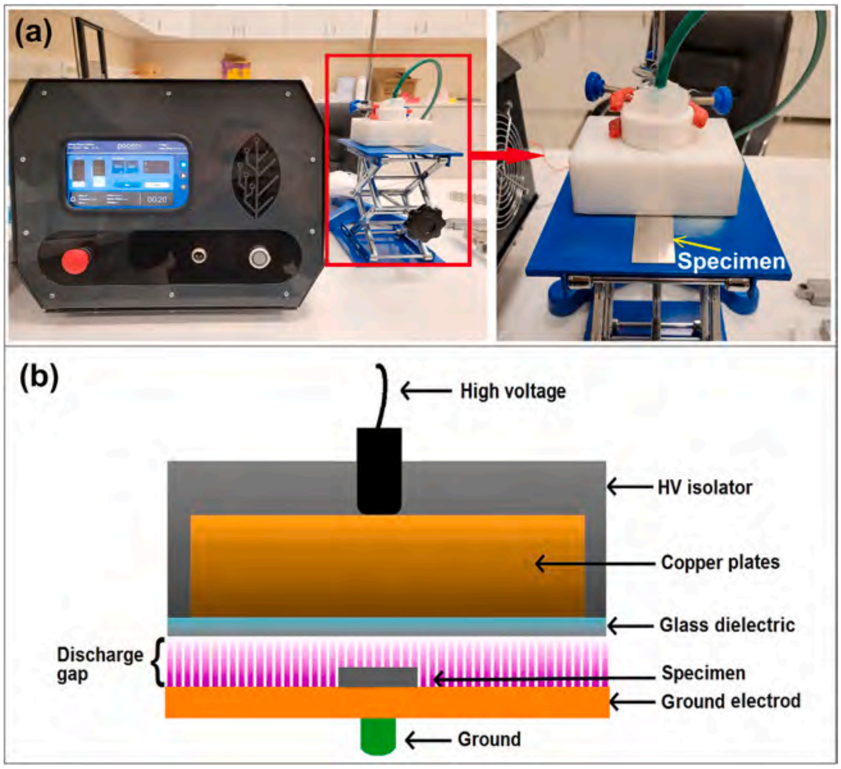

Fig. 2. (a) Atmospheric plasma surface treatment device and (b) schematic diagram of the atmospheric plasma surface treatment.

Table 4
Classification (groups) of the adherends depending on the surface treatment and the sandpaper mesh number.

No	1	2	3	4	5	6	7	8
Surface treatment	Untreated	Grinding			Plasma	Grinding + Plasma		
Sandpaper mesh number	–	P180	P320	P600	–	P180	P320	P600

(arithmetical mean roughness) is the arithmetical mean of the absolute values of the profile deviations from the mean line of the roughness profile, and R_z (mean roughness depth) is the mean value of the five greatest heights of the roughness profile values from the five sampling lengths within the evaluation length [58]. Surface roughness parameters were determined in this study in accordance with the ISO 4287:1997 standard. The surface roughness of the adherends was measured using a contact profilometer (Mitutoyo SJ-301) with a standard tracing length of 4 mm and a cut-off wavelength of 0.25 mm. The surface roughness of five aluminium alloy adherends was measured for each group given in Table 4. Three surface roughness measurements for each specimen were taken from the surface perpendicular to the grinding direction (or parallel to the loading direction for shear testing), as shown in Fig. 3(b). The average of fifteen measurements was taken to be as the ultimate result.

2.3.2. *Contact angle measurement (wettability test)*

The wettability of the surface of a material by an adhesive plays an important role in the bonding strength of a joint. The contact angle is regarded as one of the indicators of wettability. Surface treatments such as plasma and grinding have a significant influence on the contact angle on the surface of the adherend. For this purpose, the effects of grinding, plasma, and GrPl processes on the wettability of the surface of aluminium sheets were investigated by contact angle measurements in the present study. Wettability test was carried out by measuring the contact angle of a deionized water droplet on the surface of aluminium alloy adherends using a contact angle measurement analyser (Attension

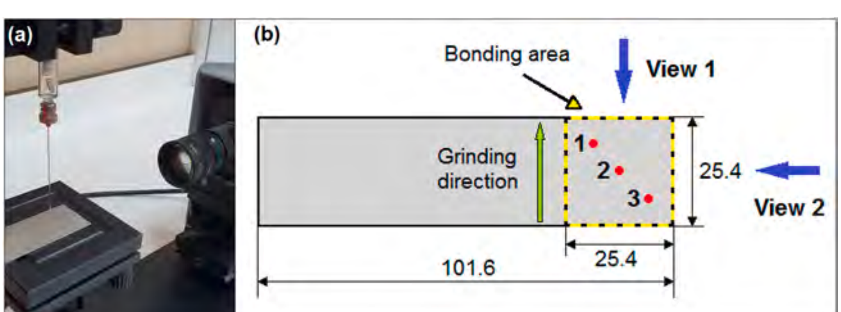

Fig. 3. (a) Contact angle measurement (wettability test) set up (b) measurement points on the aluminium sheet for contact angle and surface roughness tests (red points with the numbers 1, 2, and 3 show the measurement points).

Theta). Contact angle measurement was performed using the sessile drop method and the Young-Laplace equation. The test liquid drop volume was 4 μl, and the distance between the needle (nozzle) and the surface on which the liquid drop was placed was 4 mm. The measurements were taken at ambient humidity of 43 ± 4 % and room temperature of 25 ± 1 °C. The measuring period was 10 s. During the measuring period, the analyser conducted 12 measurements per second, and the average value of a total of 120 measurements was taken as the contact angle value. Contact angle measurements were performed in both parallel and perpendicular directions to the grinding direction. The contact angle of five aluminium alloy adherends was measured for each group given in Table 4. Three measurement points were tested on each specimen at the directions parallel (view 1) and perpendicular (view 2) to the grinding direction (Fig. 3(b)). The average of fifteen measurements was taken to be as the ultimate result.

2.3.3. XPS analysis

The chemical compositions and chemical changes in the surfaces of the untreated, plasma-treated, and GrPl-treated aluminium alloy samples were characterized with X-ray Photoelectron Spectroscopy (XPS). The aluminium sheets were cut to obtain specimens with the dimensions of 1 cm × 1 cm for XPS analysis. The specimens were inserted into the sample holder of the Thermo Scientific K-Alpha (Kα) XPS instrument using carbon tape. Afterwards, monochromatic Al Kα X-rays (1486.6 eV) with a diameter of 300 μm were directed towards the surface of each sample. Survey spectra and high-resolution spectra were obtained for each sample using pass energies of 30 eV and 200 eV, respectively. The pressure in the analysis chamber was 1 × 10^{-8} mbar. Gaussian/Lorentzian peak shapes and a Shirley/Smart type background were utilized for peak fitting.

2.4. Single-lap adhesive joints

Before manufacturing single-lap adhesive joints, aluminium alloy adherends were cleaned in an ultrasonic bath for 5 min at a frequency of 37 kHz in a solution of 10 vol.% concentrated ethanol and pure water. To prevent removing the activation of the surface of the adherends by plasma treatment, this cleaning procedure was not employed on adherends that would be treated with the plasma process.

As seen in Fig. 4, the overlap length to apply adhesive in a single-lap joint was 25.4 mm. The adhesive thickness required to bond a single-lap joint with the best (or optimum) performance is significantly dependent on the thickness of the adherend [59,60]. The thickness of adherends in single-lap joints was more than 1 mm in almost all studies found in the literature, and the influence of adhesive thickness was investigated in the range of 0.1–1 mm. As the adhesive thickness is reduced up to a critical value, the mechanical strength increases for single-lap adhesive-bonded aluminium joints [57,61,62]. However, it should also be noted that if the adhesive thickness is 0.1 mm or lower, there will be a risk of an incomplete adhesive layer between two adherends of the joint. For this reason, in the present study, the thickness of the adhesive layer was determined as 0.2 mm. The adhesive bonding process for obtaining the single-lap joints was carried out using a suitable fixture tool. In this process, firstly, adhesives were applied onto the surface of the adherends in certain amount, and then adhesive-bonded single-lap joint specimens were allowed to cure for 24 h in a fixture tool. The thickness of the

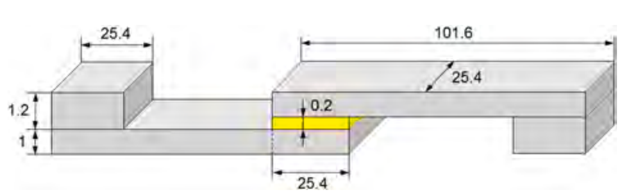

Fig. 4. Dimensions of adhesive-bonded single-lap joint specimen for shear testing.

adhesive layers in the specimens was measured as 0.2 ± 0.04 mm. There were twelve combinations to manufacture single-lap joints, including three different adhesives (cyanoacrylate, BPA-based, and BPA/F-based adhesive) and four different surface treatments (untreated, grinding, plasma, and GrPl) (Fig. 5). For each combination, five specimens were produced. Also, shear testing would be carried out at two different temperatures (room temperature and 60 °C). Thus, a total of one hundred twenty specimens were manufactured.

2.5. Shear testing

Shear tests were carried out to determine the mechanical performance of aluminium single-lap joints. In the shear test of a single-lap joint, the joint may bend because the long axis of the joint cannot coincide with the direction of the applied load. This can cause a secondary bending in the single-lap joint [63]. To minimize this probability, the load must be applied in the same direction as the centre line. In this study, the tabs were added to the end of the aluminium alloy sheets for this purpose, as shown in Fig. 6, and they were adhesively bonded to the aluminium alloy sheets using the same material as the adherend.

The shear tests were conducted in accordance with the ASTM D1002 standard at a constant crosshead speed of 1.3 mm/min. These tests were performed using a universal testing machine (Shimadzu AG-IS) at a room temperature of 25 ± 1 °C and an ambient humidity of 48 % (Fig. 6). In addition to the room temperature, the shear tests were conducted at 60 °C to investigate the mechanical performance of the adhesively-bonded single-lap aluminium joints.

For each combination of single-lap joints, five specimens were subjected to shear testing, and the average of the shear strength values of the five specimens was taken as the result. The shear strength of the single-lap adhesive joints was calculated using the following equation:

$$\tau = \frac{F}{w \bullet l}$$

where is τ the shear strength of the single-lap adhesive joint, F is the load, w is the overlap width, and l is the overlap length.

2.6. Failure analysis

Three main failure modes were identified to analyse the failures of the untreated, ground, plasma-treated, and GrPl-treated aluminium alloy specimens: adhesive failure, cohesive failure, and adhesive/cohesive (mixed) failure. The failure surfaces of the specimens were analysed at a macro level and the cross-sections of the samples were examined at a micro level using optical microscopy (Nicon-Eclipse LV150N) to investigate the relation between failure behaviour and mechanical strength in the adhesive-bonded single-lap aluminium alloy joints.

3. Results and discussion

3.1. Contact angle and surface roughness

Fig. 7 depicts the contact angles on the surfaces of untreated, ground, plasma-treated, and grinding followed by plasma (GrPl)-treated adherends. The contact angles on all of the specimens ground with P180, P320, and P600 sandpapers were smaller than that on the untreated specimens, as seen in Fig. 7(a) and (b). The contact angle on the ground specimens decreased at first and then increased as the mesh number of sandpaper increased. Contact angle values measured from the view parallel to the grinding direction (view 1) were higher than those taken from the view perpendicular to the grinding direction (view 2) for the untreated specimen, indicating that the surface roughness formed by the grinding process had a considerable influence on the contact angle (Fig. 7). The contact angles belonging to the untreated specimen were 71.8° and 65.2°, respectively, whereas the ground specimen (P320) had

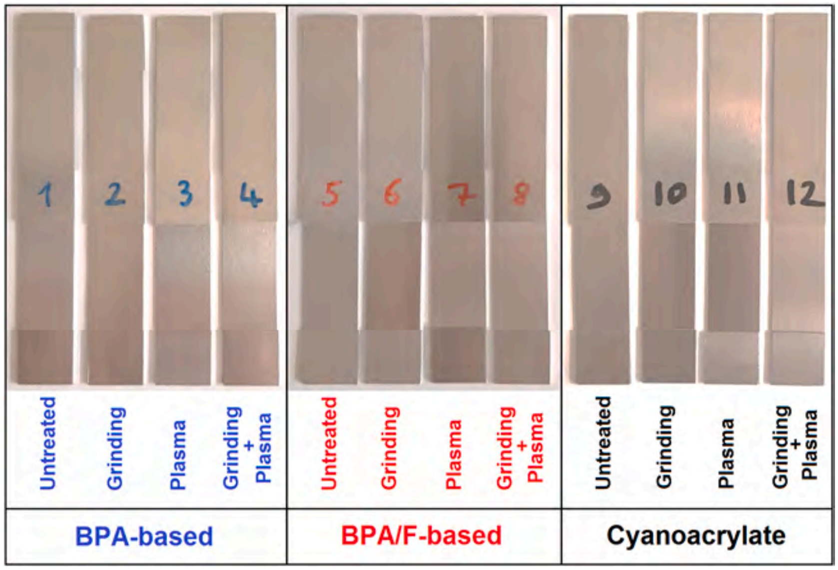

Fig. 5. Adhesive-bonded single-lap joint specimens.

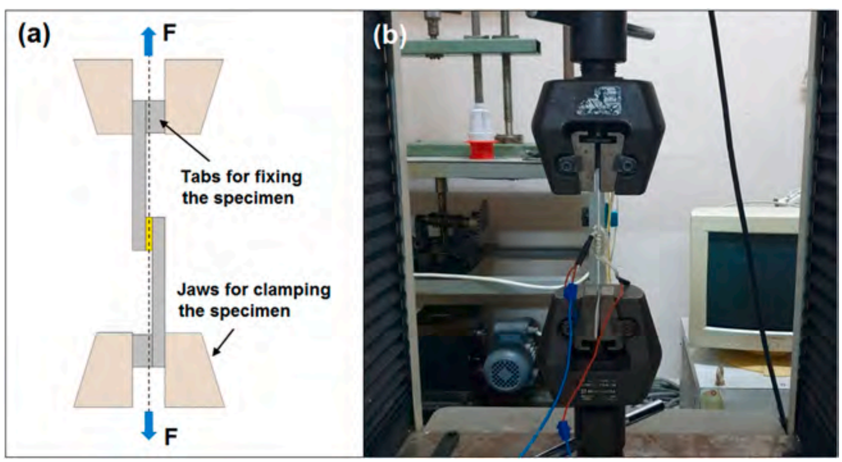

Fig. 6. (a) Load path in an adhesive-bonded single-lap joint and (b) lap-shear testing for single-lap adhesive-bonded joints.

contact angles of 55.2° and 43.5°, which increased to 58.6° and 54° for the ground specimen (P180), respectively. Liu et al. [25] observed a similar trend in contact angle depending on the mesh number of the sandpaper. Furthermore, as noted in the literature, coarse sandpapers with low mesh numbers, such as P50 and P120, result in larger contact angles [64]. The contact angle reduces as the mesh number of sandpaper increases, improving wettability and adhesion, whereas high mesh numbers over a certain mesh number do have no positive influence on wettability.

The contact angles on the plasma-treated specimens were lower than that of the untreated specimen, as seen in Fig. 7. The GrPl-treated specimens showed lower contact angles than the ground specimens. For example, while the contact angles on the specimen ground with P320 sandpaper were 55.2° and 43.5° in parallel and perpendicular directions to the grinding direction, respectively, and the application of the plasma process decreased the contact angles to 47° and 25.9°. The contact angle on the GrPl-treated specimens decreased with increasing sandpaper mesh number; however, changing the sandpaper mesh number from 320 to 180 had no influence on the contact angle. All of these results showed that the GrPl process both contributed to decreasing the contact angle on the aluminium alloy adherend, and that the plasma surface treatment had a higher effect on the reduction of the contact angle compared to grinding. The favourable effect of plasma treatment can be attributed to the fact that the plasma process cleans the surface of aluminium alloy by removing hydrocarbon contamination [65] and increases surface-free energy due to enhanced polar content of surface-free energy [42,66].

Figs. 8 and 9 show surface roughness values (R_a and R_z) and profiles of the surface of the adherends treated with grinding, plasma, and GrPl processes. The R_a and R_z of the ground specimens were higher than those of the untreated specimen and increased as the mesh number of the sandpaper decreased. The specimen ground with P180 sandpaper had a greater R_a and R_z by 3.8 and 3.1 times, respectively, than the untreated specimen (Fig. 8). Similar results were observed after the plasma treatment of ground specimens. It should be noted, however, that the plasma process had no noticeable effect on the surface roughness of the adherends, as seen in Fig. 8. The impact of ions during the plasma process can cause an increase in surface roughness [67]. The surface roughness is higher, grooves are deeper and wider, and ridges are higher and wider, resulting in improved mechanical interlocking and a greater contact area between the adherend and adhesive [67,68]. This can be achieved through a grinding process utilizing sandpapers with low mesh numbers. Grinding with a smaller mesh number, on the other hand, induces over-roughening, leading to micro-cracks in the interfacial

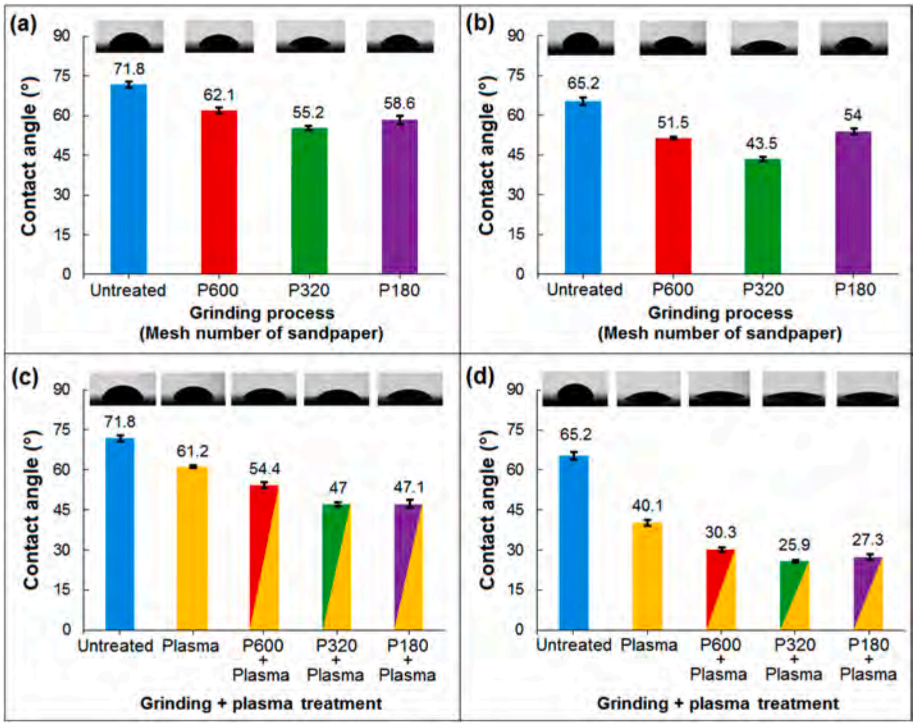

Fig. 7. Effect of the grinding process on the contact angle of the surface of the adherends in (a) parallel and (b) perpendicular directions to the grinding direction, and influence of grinding + plasma (GrPl) process on the contact angle in (c) parallel and (d) perpendicular directions to the grinding direction.

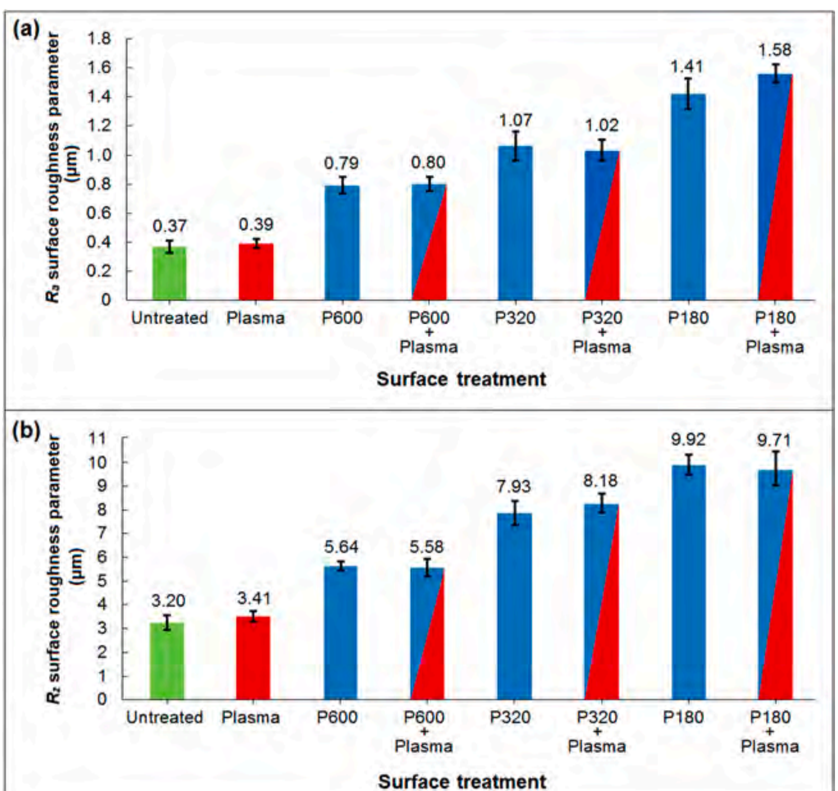

Fig. 8. Effect of surface treatments on (a) R_a and (b) R_z surface roughness parameters of aluminium alloy adherends.

region and insufficient adhesive filling into the deep grooves. Over-roughening can also cause gas bubbles to become trapped between the adhesive and the material, generating stress risers at the interface. Many of these factors reduce effective bond area and wettability, resulting in worse bonding performance, such as shear strength, of adhesive-bonded joints [25,57]. The specimens ground with P180

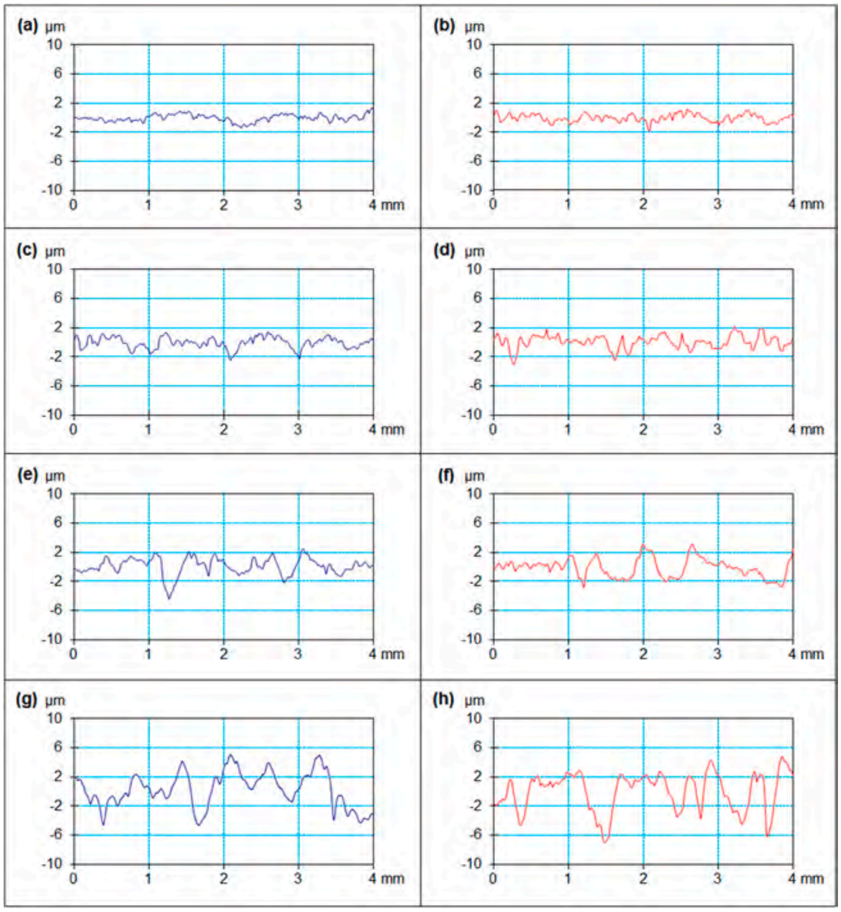

Fig. 9. Surface roughness profiles of (a) untreated, (b) plasma-treated, (c) ground (P600), (d) Gr(P600)Pl-treated, (e) ground (P320), (f) Gr(P320)Pl-treated, (g) ground (P180), and (h) Gr(180)Pl-treated aluminium alloy adherends.

sandpaper had the highest surface roughness values (Fig. 8), however, excessively deep grooves and very high ridges formed on their surfaces, indicating over-roughening (Fig. 9).

A lower contact angle on an aluminium substrate indicates higher wettability. As seen in Fig. 7, the lowest contact angle was obtained on the specimens ground with P320 sandpaper. It should be stated again that the increased surface roughness leads to deeper and wider grooves, as well as higher and wider ridges, resulting in enhanced mechanical interlocking and a greater contact area between the adherend and the adhesive. However, over-roughening can cause insufficient adhesive filling and the formation of micro-cracks, which results in a negative effect on the wettability and mechanical performance of adhesive-bonded joints. The specimens ground with P320 had a more reasonable surface roughness and profile, as observed in Figs. 8 and 9, in comparison to those ground with 180 sandpaper. As a result of these findings, the optimal sandpaper mesh number was determined to be P320, and the specimens ground with P320 sandpaper were used in the subsequent analyses of the present study.

3.2. Lap shear strength

3.2.1. Effect of surface treatment

One of the most acceptable approaches for understanding and analysing the mechanical behaviour of a single-lap joint is to evaluate its shear strength. The shear strength of a single-lap adhesive-bonded joint is affected by many factors, including the type of adhesive [69] and surface treatment applied onto the surface of the adherend [46]. Uniformity of adhesive thickness along lap length is another significant factor in the strength of a single-lap joint, which may be particularly critical in joints with a thin adhesive layer. As seen in Fig. 10, a uniform adhesive layer could be obtained in cyanoacrylate, BPA-based, and BPA/F-based adhesive-bonded joints in the present study.

Fig. 11 depicts the shear strength of adhesive-bonded single-lap joints at room temperature. Grinding and plasma surface treatments had a substantial influence on the shear strength of the joints. Grinding increased the shear strength of BPA/F-based, cyanoacrylate, and BPA-based adhesive-bonded specimens by 47 %, 54 %, and 72 %, respectively, as compared to untreated specimen. The higher shear strength of the joints produced from ground sheets can be attributed to the following factors. As shown in Fig. 7, the contact angles on the untreated specimen were 71.8° and 65.2°, and they were lowered to 55.2° and 43.5°, respectively, for the specimens ground with P320 sandpaper. A lower contact angle suggests well wettability, which is an indicator of good adhesion by physical adsorption between the adhesive and the adherend [70]. A good wetting ability allows an adhesive to spread more easily on the surface of the adherend, which contributes to improving the bonding ability between the adhesive and the adherend, leading to higher bonding strength [61,71]. With a reduction in the contact angle on the surface of the adherend ground with P320 sandpaper (Fig. 7), the lap shear strengths of the joints bonded with cyanoacrylate, BPA-based, and BPA/F-based adhesives increased (Fig. 11). It can be related to the improved wettability of the surface of the ground aluminium adherend [25]. The increase in surface roughness caused by the grinding process may contribute to the improved lap shear strength of adhesive-bonded joints formed from ground sheets, as it increases the contact area between the adhesive and the adherend. Incidentally, it should be stated that using the R_a parameter to assess the surface roughness may not be appropriate if large irregularities form on the surface of the adherend, as

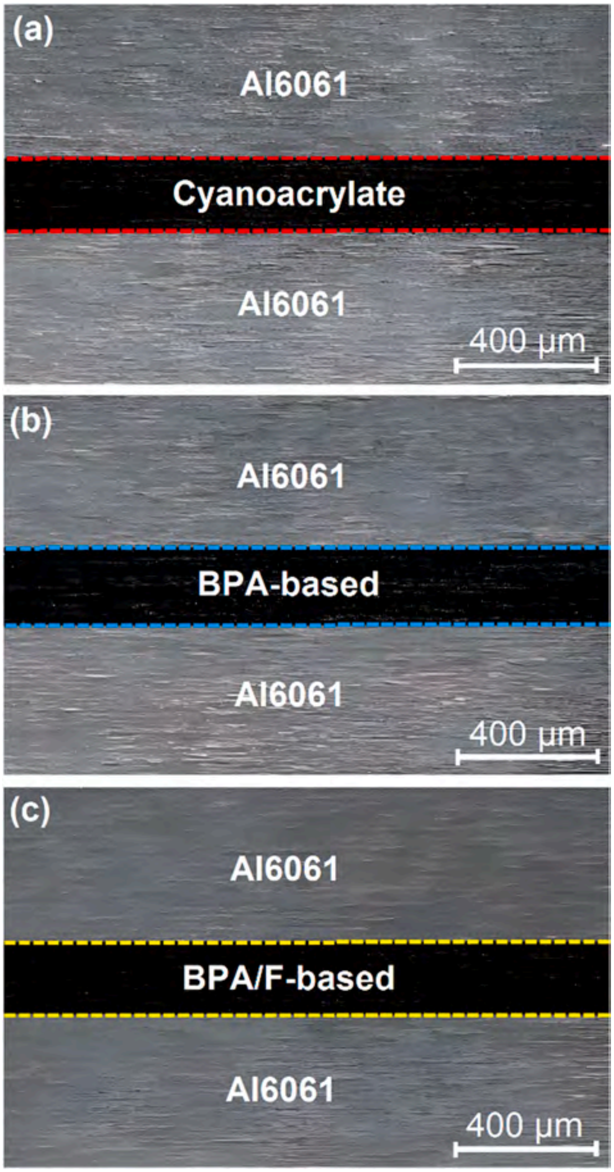

Fig. 10. Cross-section of single-lap joints bonded with (a) cyanoacrylate, (b) BPA-based, and (c) BPA-based adhesive.

depicted in Fig. 9. For this reason, the R_z parameter was used to evaluate the relation between the surface roughness and the properties of the adhesive-bonded single-lap joints. When R_z increased from 3.20 μm to 5.64 μm after grinding with P320 sandpaper (Fig. 8), the lap shear strengths of the joints bonded with BPA/F-based, cyanoacrylate, and BPA-based adhesives increased by 47 %, 54 %, and 72 % (Fig. 11). When an adherend is ground, grooves and ridges are formed on its surface. Greater surface roughness implies deeper and wider grooves, as well as wider and higher ridges. Grooves and ridges increase the contact area for bonding and the potential that the adhesive will soak into the grooves. As the height difference between the grooves and ridges increases, it will become harder to detach the adhesive from the aluminium adherend, which improves the shear strength of adhesive bonding due to the mechanical interlocking effect [25]. Compared to surface roughness profiles of the untreated specimen (Fig. 9(a)) and the specimen ground with P320 sandpaper (Fig. 9(e)), the height difference between the grooves and ridges was greater for the ground specimen, which may have contributed to the improvement of lap shear strength of the joints produced from the ground adherends. Although the grinding process improved the shear strength of the joints, it should be noted that this is

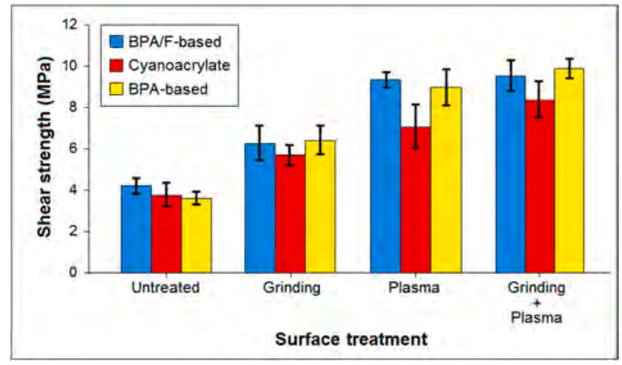

Fig. 11. Effects of surface treatments on the shear strength of single-lap joints bonded with BPA/F-based, cyanoacrylate, and BPA-based adhesives at room temperature.

only valid for a limited range of roughness values since over-roughening weakens bonding performance for the following reasons [64]. In over-roughening, the depth of grooves is significantly greater, making it difficult for the adhesive to penetrate the grooves and remove the gas from the interface between the adherend and the adhesive. This causes a reduction in the wettability of the surface of the aluminium adherend and the effect of mechanical interlocking between the adherend and the adhesive [72]. Furthermore, over-roughening may enhance stress concentration, which reduces the bonding strength of the joint [57,73]. As a result, an optimum roughening the surface of aluminium adherend has a beneficial impact on the improvement of the shear strength of the adhesive-bonded single-lap aluminium joints.

Cyanoacrylate, BPA-based, and BPA/F-based adhesive-bonded single-lap joints that have undergone to plasma treatment had higher shear strength than untreated specimens, as seen in Fig. 11. Furthermore, the plasma process improved lap shear strength more than the grinding process. This can be related to the fact that the plasma process cleans and activates the surface of the aluminium alloy by raising surface free energy and generating adhesion-promoting surface functional groups [36]. The plasma process provides a favourable condition for the formation of active hydroxyl groups, which enhances surface free energy or reduces contact angle of the surface of the aluminium alloy by increasing the polar component of the surface energy [51,74,75]. The contact angle on the surface of the adherend was lowered when the plasma process was applied to its surface, as shown in Fig. 7, which may have contributed to an improvement in the lap shear strength of the joints. Plasma treatment increased the shear strength of BPA/F-based, cyanoacrylate, and BPA-based adhesive-bonded joints by 122 %, 89 %, and 149 %, respectively.

The atmospheric pressure plasma process favours the growth of an oxide layer on the surface of the aluminium. XPS analysis was performed to identify oxide-like components and metallic aluminium on the surfaces of plasma-treated and untreated samples. As shown in Fig. 12, while there was no significant change in the amount of metallic aluminium, there was an increase in the amount of oxygen after the plasma treatment. In addition, the binding energies of the oxide groups decrease after plasma treatment, indicating that hydroxyl groups are also formed on the surface. As a result of their polar nature, these oxide and hydroxyl groups detected in the surface composition have an increasing effect on the wettability of aluminium. Moreover, hydroxyl groups can enhance the strength of interface by facilitating the formation of hydrogen bonds with the adhesive [76]. XPS analysis with the C-1s spectrum was performed on untreated and plasma treated surfaces to examine oxides and similar components in greater detail. There was no significant difference in the amounts of C–C/C–H and C–O components in these two samples. On the other hand, the C═O component identified on the untreated sample surface was oxidized by plasma to form the COOH component and thus was not detected on the plasma

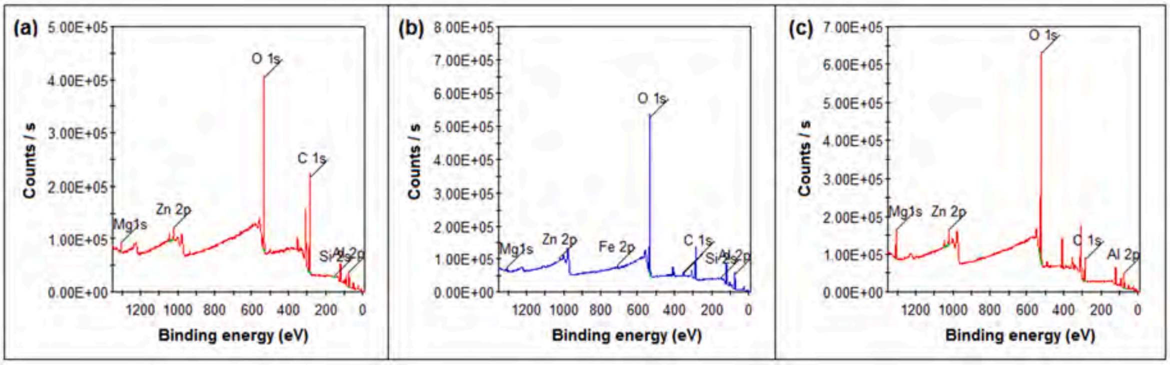

Fig. 12. XPS spectra for the surfaces of (a) untreated, (b) grinding + plasma-treated, and (c) plasma-treated aluminium alloy.

treated sample surface (Fig. 13). The presence of COOH, a hydroxyl group, in the structure increased the polarity of the surface, which improved its wettability. Furthermore, the ability of COOH to form hydrogen bonds strengthened the interaction between the adhesive and aluminium. Meanwhile, polar hydroxyl groups on aluminium oxide can promote strong chemical bonding between hydroxyl groups and molecules in an adhesive such as epoxy, which improves the adhesion between the adherend and the adhesive [77,78].

Cyanoacrylate, BPA-based, and BPA/F-based adhesive-bonded joints treated with GrPl had higher shear strength than those treated with only grinding or plasma processes (Fig. 11). It can be due to the synergistic effect of the aforementioned favourable influences of grinding and plasma processes on the shear strength of adhesive-bonded single-lap joints.

Fig. 14 depicts the shear strength of single-lap joints at 60 °C. When the testing temperature was raised from room temperature to 60 °C, the bonding performances of all adhesive-bonded joints deteriorated. The shear strength of BPA/F-based, cyanoacrylate adhesive, and BAP-based adhesive-bonded joints made of untreated adherends decreased by 13 %, 21 %, and 42 %, respectively. It can be attributed to a reduction in the strength of the adhesives caused by temperature rise. Similar behaviour was observed in the adhesive-bonded joints produced from the plasma-treated sheets (Fig. 14). However, while the grinding process had a significant beneficial influence on the shear strength of joints at room temperature, it had a considerable deteriorating effect at 60 °C in comparison to the plasma treatment effect. As the temperature rises, the adhesive gets more ductile [79] and the mobility of the polymer chains in the adhesive increases [80], which allows the adhesive to deform plastically and the stress to redistribute, resulting in less stressed regions of the joint [81]. According to this phenomenon, there should not have been such a substantial reduction in the shear strength of the joints made of ground sheets at 60 °C. However, it can be suggested that stress concentration caused by surface roughness generated during the grinding process was still an important factor at this temperature, and the combined negative effects of temperature rise and stress concentration may have led to the strength of the joints made of ground adherends to further declining. Meanwhile, it should be noted that even though the joints manufactured from the ground sheets experienced the highest reduction in shear strength due to temperature increase, these joints still had higher strength than those made of untreated sheets (Fig. 14). While the grinding process had a positive impact on the shear strength of the adhesive-bonded joints at 60 °C, it was less pronounced than it had been at room temperature; however, the plasma treatment had a greater positive impact than the grinding process at 60 °C.

3.2.2. *Effect of adhesive type*

The type of adhesive has a considerable influence on the bonding performance of adhesive-bonded single-lap joints. Comparing the lap shear strengths of cyanoacrylate, BPA-based, and BPA/F-based adhesive-bonded joints produced from untreated adherends, the BPA/F-based adhesive-bonded joint yielded the highest strength while the joint bonded with BPA-based adhesive had the lowest shear strength (Fig. 11). The highest lap shear strength, however, was achieved for BPA-based adhesive-bonded joint made of ground adherends. It can be attributed to the fact that there is a strong relationship between the viscosity of a liquid and its ability to wet the surface of a solid [82], and a liquid with low viscosity spreads over the surface of a solid and infiltrates cavities on its surface. Among the adhesives investigated in this study, cyanoacrylate adhesive has the lowest viscosity whereas BPA/F-based adhesive has the highest viscosity (Table 3). Based on this

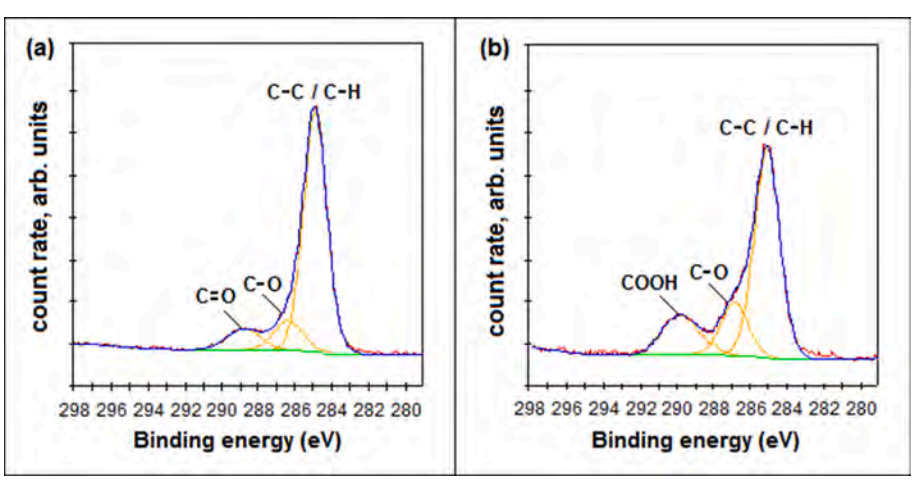

Fig. 13. XPS C-1s spectra for the surfaces of (a) untreated and (b) plasma-treated aluminium alloy.

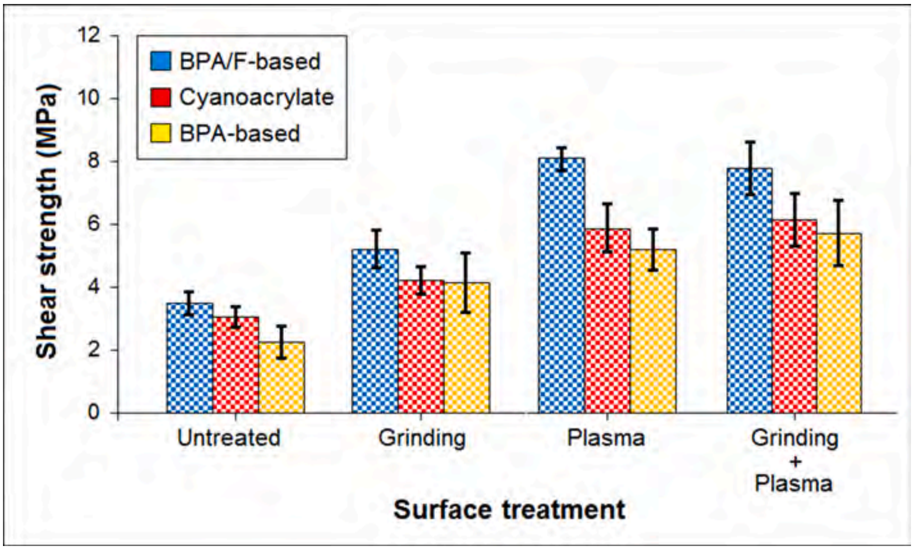

Fig. 14. Effects of surface treatments on the shear strength of single-lap joints bonded with BPA/F-based, cyanoacrylate, and BPA-based adhesives at 60 °C.

phenomenon, it would be expected that cyanoacrylate adhesive spreads more easily over the surface and fills the grooves than BPA/F-based adhesive. This would lead to a higher contact area and better mechanical interlocking between the cyanoacrylate adhesive and the adherend, which would result in higher shear strength for the cyanoacrylate adhesive-bonded joints than the BPA/F-based adhesive-bonded joints. In the present study, however, when BPA/F-based adhesive and cyanoacrylate adhesive-bonded joints made of untreated and ground adherends were evaluated, the opposite results were obtained, as seen in Fig. 11. As a result of these findings, it can be concluded that the bonding performance of these joints should be attributed not only to the contact area and mechanical interlocking but also to other properties of the adhesives, such as the ability to form chemical bonds with the aluminium alloy adherend depending on the specific (different) chemical compounds of each adhesive (Table 3).

Although the BPA/F-based adhesive-bonded joint showed higher shear strength than the BPA-based adhesive-bonded joint for single-lap joints manufactured from untreated sheets, the BPA-based adhesive-bonded joint had better bonding performance than the BPA/F-based adhesive-bonded joint when grinding process was applied to the surface of the adherends. The grinding process may result in the creation of deep grooves on the surface depending on the mesh number of sandpaper. Because it has a lower viscosity than BPA/F-based adhesive, the capillary wetting mechanism in the adhesive made of BPA-based epoxy and hardener may have been more efficient. It can be attributed to the fact that this adhesive with low viscosity may have flowed more easily into the grooves driven by the capillary force [83], potentially increasing the effective contact area and mechanical interlocking between the epoxy adhesive and metal adherend [84]. As a result, the shear strength of the single-lap joint manufactured by bonding the ground adherend with BPA-based adhesive was higher than that of the BPA/F-based adhesive-bonded joint. The findings suggest that the type of adhesive used affects the shear strength of single-lap joints manufactured of untreated sheets, depending on the adhesive characteristics such as adsorption and strength. When the surface of the adherend is treated to a grinding process, adhesive properties such as viscosity have a substantial impact on the shear strength of joints due to increased effective contact area and mechanical interlocking.

BPA/F-based and BPA-based adhesive-bonded joints had significantly higher lap shear strength than cyanoacrylate adhesive-bonded joints when plasma treatment was applied to the surface of the adherends (Fig. 11). As previously stated, hydroxyl groups generated by plasma treatment on the surface of aluminium can promote strong adhesion bonding with molecules in epoxy, improving adhesion between the surface of the adherend and the adhesive. The higher shear strength of BPA/F-based and BPA-based epoxy adhesive-bonded joints produced from plasma-treated sheets can be related to this factor.

Higher shear strength values were obtained for the joints made of ground and plasma-treated sheets when the adhesive with BPA and BPA/F were used, respectively, whereas the highest shear strength was achieved for the joint produced from GrPl-treated sheets when the BPA-based adhesive with low viscosity epoxy resin was used (Fig. 11).

Fig. 14 illustrates the shear strength of single-lap joints at 60 °C. When the testing temperature increased from room temperature to 60 °C, the shear strength of single-lap joints bonded using different adhesives decreased compared to those at room temperature (Fig. 11). The highest shear strength values at 60 °C were achieved when the untreated, ground, and plasma-treated sheets were bonded using BPA/F-based adhesive. While the BPA-based adhesive with low-viscosity resin had the most beneficial impact on improving the shear strength of the joints at room temperature, the opposite result was obtained at 60 °C. It may be related to the change in the properties such as the strength of the adhesive at high temperatures [84,85]. These findings could indicate that the bonding performance of the single-lap joints was dominated by the substantial reduction in the strength of the BPA-based adhesive at 60 °C.

All of the results revealed that, at room temperature, the BPA-based and BPA/F adhesives were more effective on shear strength of single-lap joints than cyanoacrylate adhesive and that BPA/F-based adhesive was the best adhesive for bonding untreated and plasma-treated sheets, while BPA-based adhesive was the best for bonding ground and GrPl-treated sheets. At 60 °C, BPA/F-based adhesive had the most favourable influence on the shear strength of all joints, whereas joints bonded with BPA-based adhesive had the least shear strength.

3.2.3. Failure behaviour

In adhesive-bonded single-lap joints subjected to shear testing, three types of bond failure primarily occur: adhesive failure, cohesive failure, and adhesive-cohesive failure [86]. Adhesive failure is an interfacial failure that occurs at the interface between the adherend and the adhesive, resulting in complete separation of the adhesive from the surface of one of the adherends. Cohesive failure takes place in the adhesive itself, and both surfaces of the adherend remain covered with the adhesive. Bond failures can occur in two modes (mixed failure mode), which include both adhesive and cohesive failure. Adhesive failure occurs when the interfacial strength between the adhesive and the adherend is weaker than the strength of the adhesive, whereas cohesive failure takes place in the opposite circumstance. Analysis of the failure modes of adhesive-bonded single-lap joints is one of the most important ways to comprehend and evaluate the bonding performance of these joints [49,87,88]. Fig. 15 depicts the failures that occurred in the adhesive-bonded single-lap joints subjected to shear testing at room temperature. In the joints bonded with untreated sheets, adhesive failure took place (Fig. 15(a)–(c)). Compared to a ground sheet, an untreated sheet would have a lower roughness. The lower the surface roughness is, the less mechanical interlocking will be, which will lead the adhesive adhering to the metal surface to slide easily and the crack to propagate promptly along the interface [84]. This may have caused the cyanoacrylate, BPA-based, and BPA/F-based layers to completely separate from the surface of one of the adherends in the joints that were manufactured from untreated (no grinding) adherends (Fig. 15(a)–(c)). When the surfaces of the sheets were ground, adhesive, partial-adhesive, and mostly cohesive failures were the main failure modes for BPA/F-based, cyanoacrylate, and BPA-based adhesive-bonded joints, respectively (Fig. 15(d)–(f)). The higher the surface roughness is, the higher the edges and the deeper the grooves are, resulting in more contact areas and mechanical interlocking between the adhesive and the adherend than on smoother surfaces, which contributes to better adhesion in adhesive-bonded single-lap joints [57]. While the grinding process had a beneficial effect on improving the shear strength of adhesive-bonded single-lap joints the highest shear strength was achieved for the BPA-based adhesive-bonded joint manufactured from ground sheets (Fig. 11). In the cross-section of BPA-based adhesive-bonded joints produced from ground sheets prior to shear testing, no voids were observed in the grooves or on the surface of the joint, as seen in Fig. 16(a). It can be attributed to the fact that epoxy such as BPA-based adhesive promotes greater interfacial strength due to more rapid and full penetration into the micro-voids [70]. Both the presence of grooves and ridges and the absence of voids on the surface of the adherend lead to a greater effective contact area and mechanical interlocking. As the effective contact area expands, stress concentration reduces, resulting in adhesive-bonded joints with improved shear strength [73]. Furthermore, mechanical interlocking provokes energy expenditure during fracture, which effectively constitutes adhesion strength [84]. As seen in Fig. 16(a), mechanical anchoring between the surface of the aluminium alloy adherend and the adhesive took place in the BPA-based adhesive-bonded joint manufactured from ground sheets. All of these factors contributed to the highest shear strength obtained in the BPA-based adhesive-bonded joint made of ground adherends. If the grooves and valleys on the surface of the adherend are not completely filled with adhesive due to a lack of wetting and a gas entrapment between the adherend and adhesive, the effective contact area will be reduced, stress will be concentrated at the interface more, and the shear strength of the adhesive-bonded joints will subsequently decrease [57].

A mixed mode failure occurred in the joints produced from plasma-treated sheets, as seen in Fig. 15(g)–(i). As previously stated, molecules in epoxy and hydroxyl groups created by plasma treatment on the surface of aluminium are compatible with each other in order to form enhanced adhesion bonding. No discontinuity/defect was observed at the interface between the adherend and the adhesive in the joints made of plasma-treated sheets (Fig. 16(b)), which would suggest that the interfacial bonding was strong. The shear strength of adhesive-bonded joints, both BPA-based and BPA/F-based, produced from plasma-treated sheets, demonstrated enhanced performance. However, the BPA/F-based adhesive-bonded joints exhibited slightly greater shear strength compared to their BPA-based joints. It can be attributed to partial cohesive failure observed in the fracture surface of BPA/F-based adhesive-bonded joint made of plasma-treated sheets (Fig. 15(g)),

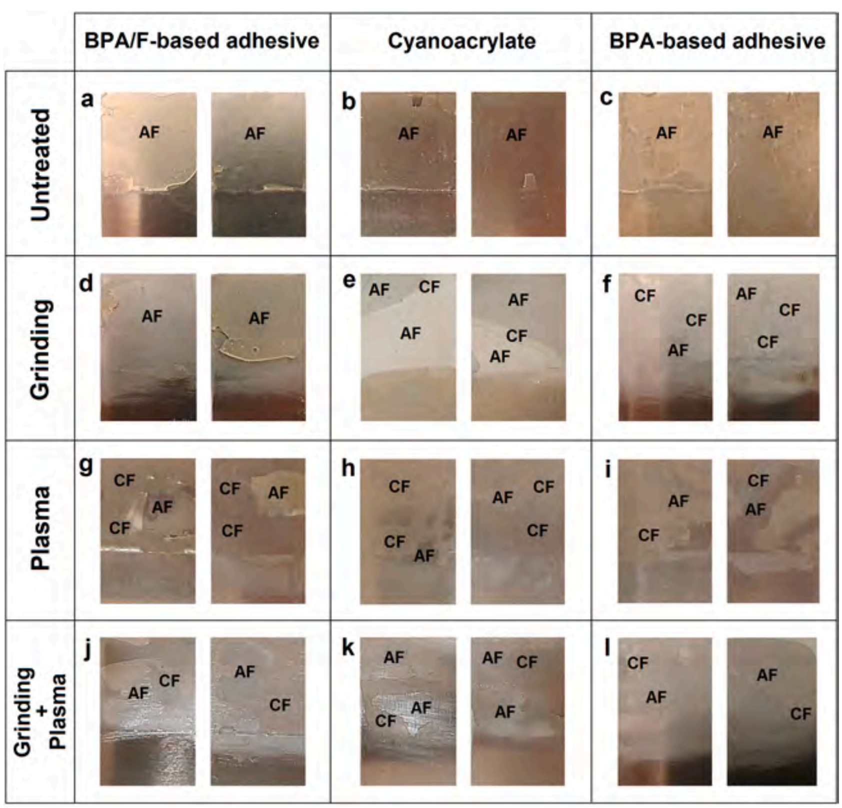

Fig. 15. Failure surfaces of the single-lap joints treated with various surface treatments and bonded with different adhesives at room temperature (a) untreated−BPA/F-based, (b) untreated−cyanoacrylate, (c) untreated−BPA-based, (d) grinding−BPA/F-based, (e) grinding−cyanoacrylate, (f) grinding−BPA-based, (g) plasma−BPA/F-based, (h) plasma−cyanoacrylate, (i) plasma−BPA-based, (j) GrPl−BPA/F-based, (k) GrPl−cyanoacrylate, and (l) GrPl−BPA-based. (AF: Adhesive Failure and CF: Cohesive Failure).

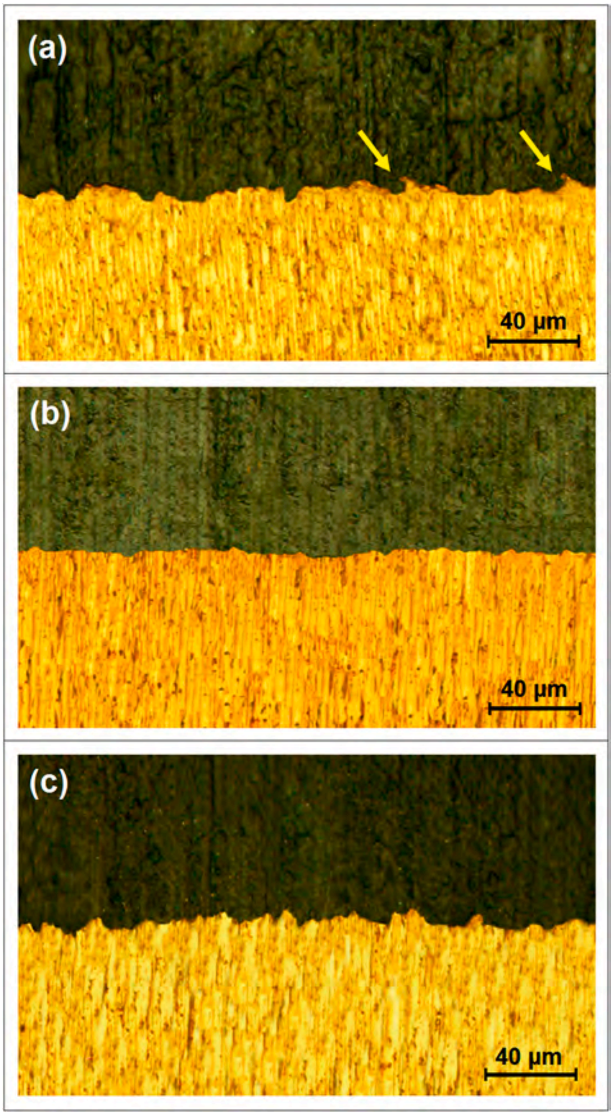

Fig. 16. Optical images of the cross-sections of single-lap joints with various surface treatments and adhesive types: (a) grinding−BPA-based adhesive (yellow arrow shows mechanical anchoring between the surface of the aluminium alloy adherend and the adhesive), (b) plasma−BPA/F-based adhesive, and (c) grinding followed by plasma−BPA-based adhesive.

because partial cohesive failure mode results in considerable energy dissipation during fracture [84].

The joints manufactured from GrPl-treated sheets experienced mixed failure mode (Fig. 15(j)–(l)). Similar to the joints produced from plasma-treated sheets, no failure was observed at the interface between the adherend and the adhesive in the joints made of GrPl-treated sheets (Fig. 16(c)). This indicates the positive synergistic effects of grinding and plasma processes on the bonding performance of the single-lap joints, considering the strength values of the joints.

Fig. 17 depicts the failures that occurred in the adhesive-bonded single-lap joints subjected to shear testing at 60 °C. As the temperature increases, the strength of an adhesive diminishes, while its ductility increases, leading to a significant amount of plastic deformation in the adhesive [85]. Because of this reason, the adhesives in the joints subjected to shear testing at 60 °C deformed plastically considerably, resulting in cohesive failure in the adhesives (Fig. 17). Due to the greater bond strength between the adhesive and the surface of the ground adherend compared to the surface of the untreated adherend, the fracture is caused by a higher degree of plastic deformation within the adhesive. This results in increased fracture energy and consequently higher strength in the joints made of ground sheets. A cohesive failure occurred in the BPA-based adhesive-bonded joint (Fig. 17(b)) while the BPA/F-based adhesive-bonded joint had mixed failure mode (mostly cohesive failure) (Fig. 17(a)). It should be noted that while the BPA-based adhesive-bonded joints had higher shear strength at room temperature than the other adhesives used in the present study (Fig. 11), BPA/F-based adhesive-bonded joints had the highest shear strength at 60 °C (Fig. 14).

4. Conclusions

The effects of various surface treatments (grinding, plasma) and different adhesive types (cyanoacrylate, BPA-based, and BPA/F-based adhesives) on the bonding performance of single-lap joints produced from thin Al6061-T4 sheets at room temperature and 60 °C were investigated. The following conclusions can be drawn.

- While the contact angle on the surface of the untreated adherend was 71.8°, it was reduced to 55.2° and 43.5° in parallel and perpendicular directions to the grinding direction, respectively, when the surface of the adherend was ground using P320 mesh sandpaper. Grinding followed by the plasma process on the ground surface reduced the contact angle to 47° and 25.9° in parallel and perpendicular directions, respectively.
- Grinding and plasma treatments considerably enhanced the lap shear strength of cyanoacrylate, BPA-based, and BPA/F-based adhesive-bonded single-lap joints. As compared to the joints produced from untreated adherends, the lap shear strengths of BPA/F-based, cyanoacrylate, and BPA-based adhesive-bonded joints manufactured from ground sheets improved by 47 %, 54 %, and 72 %, respectively.
- After plasma treatment of the surface of the adherends, the shear strength of BPA/F-based, cyanoacrylate, and BPA-based adhesive-bonded joints increased by 122 %, 89 %, and 149 %, respectively,

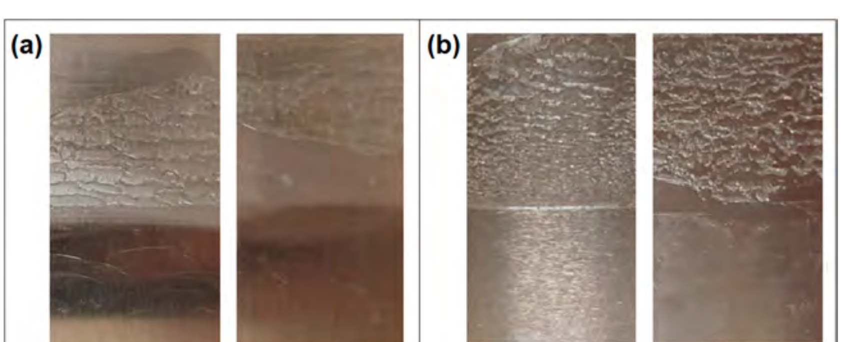

Fig. 17. Failure surfaces of the adhesive-bonded single-lap joints at 60 °C: (a) grinding−BPA/F-based adhesive and (b) plasma−BPA-based adhesive.

compared to joints produced from untreated adherends. The synergistic impact of grinding followed by plasma treatment resulted in a 126 %, 123 %, and 174 % enhancement in shear strength of BPA/F-based, cyanoacrylate, and BPA-based adhesive-bonded single-lap joints, respectively.

- When the temperature was raised from room temperature to 60 °C, the bonding performances of all single-lap joints deteriorated due to the decrease in the strength of the adhesive. The grinding process slightly increased the shear strength of the joints at 60 °C, although not as well as at room temperature; however, plasma treatment had a more beneficial effect on improving the shear strength of the joints than the grinding process.
- The type of adhesive significantly affected the lap shear strength of joints manufactured from untreated, ground, and plasma-treated adherends. High shear strength values were obtained for the joints made of ground and plasma-treated adherends when BPA-based and BPA/F-based adhesives were used, respectively, whereas the highest shear strength was achieved for the joint produced from GrPl-treated adherends when the BPA-based adhesive was used.
- When the testing temperature increased from room temperature to 60 °C, the shear strength of single-lap joints bonded using different adhesives decreased. The highest shear strength values at 60 °C were achieved for the joints bonded with BPA/F-based adhesive. While the BPA-based adhesive had the most beneficial impact on improving the shear strength of the joints at room temperature, the opposite result was obtained at 60 °C. It may be related to the change in the properties such as the strength of the adhesive at high temperatures.
- The adhesive (interfacial) failure occurred in BPA/F-based, cyanoacrylate, and BPA-based adhesive-bonded joints produced from untreated adherends. When the surface of the adherend was ground, adhesive and partial adhesive failures were the main failure modes for BPA/F-based and cyanoacrylate adhesive-bonded joints, respectively, while cohesive failure was the dominating failure mode for the BPA-based adhesive-bonded joint. Mixed failure mode occurred in the joints produced from the plasma and GrPl-treated adherends. When the joints were subjected to shear testing at 60 °C, cohesive failure occurred as a result of the decrease in the strength and the increase in deformation of the adhesives.

CRediT authorship contribution statement

Ege Gülçiçek: Writing – review & editing, Writing – original draft, Methodology, Investigation, Data curation. **Ege Anıl Diler:** Writing – review & editing, Writing – original draft, Validation, Supervision, Methodology, Investigation, Data curation, Conceptualization. **Onur Ertugrul:** Writing – review & editing, Writing – original draft, Validation, Supervision, Project administration, Methodology, Investigation, Funding acquisition, Data curation, Conceptualization.

Declaration of competing 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.

Data availability

Data will be made available on request.

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