Influence of Surface Roughening of the Raw Material on the Lap Shearing Strength and Failure Behavior of Adhesively Bonded Aluminum Joints

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Influence of surface roughening of the raw material on the lap shearing strength and failure behavior of adhesively bonded aluminum joints

Jianpeng Liu · Wei Wang · Yong Yan · Hong He · Zhigang Xue · Congchang Xu · Luoxing Li

Abstract

Surface roughening of the substrates before bonding plays a significant effect on improving the mechanical performance of the adhesively bonded joints, which are prevalently used for light-weighting vehicle bodies. In this study, the influence of surface roughening on the lap shearing strength and failure behavior of adhesively bonded aluminum sheet joints was investigated. Sandpaper grinding was employed for surface roughening, methods such as tensile testing microstructure observation, etc., was employed for evaluating the performance of the joints. The results showed that the lap shearing strength of adhesively bonded joints increased and then decreased with the surface roughness of the aluminum substrate. The maximum shearing strength of the joint bonded with grinded substrates was 30.4 MPa which was improved by 57.5% compared to that produced with un-grinded substrates. However, over roughening is harmful. When the surface roughness was too large, the failure mode of the joint turned from the mixed failure mode to interfacial failure mode, which decreased the strength of the joints. Related mechanisms were demonstrated. When the substrate surface was

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coarsened, the bonding area and the wettability of the adhesive on the surface were both increased, which promoted the beneficial mechanical interlock effect between the adhesive and the substrate. However, when the substrate surface was over roughened, defects such as voids, insufficient infiltrating of the adhesive, etc., were induced, which apparently increased the proportion of the interfacial failure area.

**Keywords:** Surface treatments; Wettability; Bonded joints; Failure behavior; Interfacial adhesion; Lap shearing strength

1 Introduction

Adhesively bonded aluminum structural parts got potential applications in recent years in the design of advanced transportation tools, such as automotive, aircraft and marine applications. Their exceptional performances include light-weighting, sealing, sound insulating, high stiffness and corrosion resistance, etc. Compared to welding, riveting and bolting, the residual stress and micro-cracks in the adhesively bonded joints are much less. The operability and efficiency of the adhesive bonding technology are much higher. For example, it can be operated in a narrow space without sophisticated equipment and can be fully cured along with the paint baking process which is very cost-effective. Therefore, the performances of such bonded and multilayered joints have attracted great attention [1-5].

The mechanical strength and failure behavior under diverse service conditions are the most concerning issues for the adhesively bonded aluminum joints, especially when they are used in vehicle bodies. The dangerous points should be known when engineers design and verify the safety of the structure. They are mainly depended on the cured
adhesive strength, and the bonding interface quality in application which are sensitively influenced by the processing parameters. Many factors are involved, such as the surface state of the adherend, the thickness of the adhesion layer, etc. Among them, the surface roughness of the aluminum substrate to be bonded is the most important. In the early studies as reported in the literature, researchers found the shear strength of the adhesively bonded joints increased with the surface roughness of the metal sheet. Shahid et al. tested the strength of cleavage joints metal sheets with various surface roughness, and found that the cleavage joint strength has a monotonic relationship with the surface roughness of the raw materials [6]. However, the results were examined in a small surface roughness range. Generally, engineers usually prefer to design single lap joints during manufacturing of automobiles and it is essential to know the lap shearing strength of adhesively bonded joints in the scope of a larger surface roughness. Several reports pointed out that there exists a most suitable surface roughness for the raw materials which bring about maximum tensile strength of the adhesively bonded aluminum joints [7-10]. Tezcan studied the influence of surface roughness on the adhesively bonded joints strength under impact loading with different strain rates. It was demonstrated that the lap shearing strength of the adhesively bonded joints will be a minimum value when the surface roughness $R_a$ of the substrate was less than 0.1 μm or higher than 2.5 μm [11]. Ghumatkar et al. treated the surface of adherend materials with the different mesh numbers of sandpaper to obtain different parameters of roughness, and found that the maximum strength of aluminum/steel adhesive joints corresponds to the most appropriate surface roughness values. When the surface roughness $R_a$ of the
aluminum substrate and the steel substrate were 2.05 μm and 1.98 μm. The maximum
strength of the adhesively bonded joints was 4.97 MPa and 6.78 MPa [12]. These
studies help engineers to realize that the relevance between the lap shearing strength of
the adhesively bonded joints and the surface roughness of the raw materials is not
linear. Nevertheless, the related mechanism of this phenomenon is still indistinct.

Since surface roughening of the substrate to some extent plays an active effect in
strengthening the mechanical behaviors of the adhesively bonded joints, researches
were further conducted to study the influence of the surface morphology. A quantity of
surface treatment methods was attempted to obtain different surface status, including
sandpaper grinding, plasma etching, sandblasting, chemical etching, anodizing, etc.
Through these, different types of grooves were obtained, which were found to be
effective in reducing stress concentration and improving the adhesion quality by
preventing entrapped air at the interface. Using sandpapers with different mesh
numbers, Ghumatkar et al found that the surface roughness for aluminum substrates
could be achieved in a wide range by the various grinding direction and duration [12].
Da Silva et al verified the influence of grooves and scratches on the joint strength with
brittle adhesives and ductile adhesives comparatively. The results showed that scratches
or grooves produced by grinded aluminum substrate were harmless and even increase
interface force [13]. Wong et al. modified the surface of aluminum substrates using a 20
Hz Nd-YAG pulsed laser and generated an aperiodic concentration ring with constant
depth and diameter [14]. The results demonstrated that the bonding quality of the
adhesive joints was promoted apparently. Among the above methods, mechanical
technology, sandpaper grinding, for example, is still considered to be optimal for large-scale industrial production, in view of the operability, efficiency, and economic cost.

Clarifying the mechanisms for the strengthening of the interface of the bonded metal joints through surface treatment of the raw material is crucial for the processing parameters designing. In the study carried out by Ghumatkar et al, they hold the opinion that the improved adhesion quality resulted from mechanical interlocking between the adhesive layer and metal surface [12]. Dong-Jun Kwon et al. investigated the influence of the surface roughness on the interfacial adhesion strength of joints. Experimental results showed that the mechanical anchoring effect of the joints was enhanced by increasing the roughness of the aluminum surface [15]. J.P.B. van Dam et al. investigated the influence of surface roughness on the interfacial adhesion of epoxy adhesively bonded steel joints through different surface treatments. They revealed that surface roughening has a significant impact on the strength in the joints tensile test and the improved shear strength mainly resulted from the increased interfacial bond area [16]. Even though, regarding the mechanisms, the consensus has not been reached. The reason is the adhesive layer itself and the bonding surface are always the weakest part. Furthermore, the dangerous points largely affect the strength of adhesive joints, for example the adhesive layer, the adhesive interface and a mixed mode. The above studies showed results of the increase in strength of adhesively bonded joints by surface roughening, but they did not point out the failure behavior and the strengthening mechanisms.
Different treatment types of adhesively bonded joints exhibited different failure modes under tensile or shear load conditions. Pietro Maressa et al. studied failure mechanisms of the adhesively bonded Ti6Al4V sheets with laser processed patterns on the surface, and discovered partial cohesive failure, fully cohesive failure and textured substrates failure in various laser treatments [17]. Chen et al. investigated the damage characteristics and failure behavior of hybrid materials joints, and reported that the adhesive joints showed a combined mode of fiber-tear failure, adhesive failure and cohesive failure [18]. The failure of the bonding surface can be prevented by roughening the substrate surface. Surface chemical modification has also been proved to be an available way to improve the bonding strength because the bonding state during the adhesively bonded joint preparation process is involved. Surface roughening provides the beneficial mechanical interlocking between the aluminum substrate and adhesive, but over roughening would reduce the wettability of the substrates thus the strength of the joints was weakened. Voids possibly occur at the bonding interface in this condition, which accelerates the crack propagation and lowers the strength of the interfacial force.

For example, the influence of surface characteristics on the strength of adhesively bonded aluminum joints was investigated by Boutar, and found that the bonding quality decreases with increasing surface roughness [22]. In the study carried out by Yang [23], it found that non-permeable zones, an alternation of solid-liquid-gas interfaces were produced if the substrate was over roughened which were both harmful to the bonding quality.
From the results reported in the literature, it can be summarized that the mechanical strength and failure behavior should be taken into consideration together when engineers evaluate the performance of an adhesively bonded joint. Unfortunately, such comprehensive researches are still few. It triggers our present study. Different surface roughness was produced on the bonding surface of aluminum sheets by grinding with sandpapers of different mesh number. The surface roughness, morphology and free energy, wettability of the adhesive on the aluminum substrates with or without roughening, lap shearing strength and interface failure models of the adhesively bonded joints under shear loading were measured and compared. The contact angle test equipment was used to investigate the relationship between the surface roughness and surface free energy. The influence of surface roughness, microcosmic area and surface free energy on the lap shearing strength was examined. The related mechanisms for the failure behavior of adhesively bonded joints were systematically discussed.

2 Materials and methods

2.1 Raw materials

A commercial 6063 aluminum alloy in the state of the extruded sheet was used as the raw material, which has excellent mechanical performance and is widely used in the field of automobile manufacturing. Specimens with sizes of 100 mm in length, 3.8 mm in thickness and 25 mm in width were cut by a wire-cut electric discharge machine for adhesive bonding. A structural adhesive for the automobile industry, Dow BETAMATE 1840C, a single component, heat-cured epoxy was employed to the adhesion. Its density
is 1.24 g/ml at 23 °C, and the viscosity is 46 Pa.S at 45 °C. Its tensile strength is 37.1 MPa.

2.2 Surface treatment of the aluminum substrates

Sandpaper grinding was adopted as the surface treatment of the aluminum sheets, which were subdivided into six groups to investigate the mechanical behaviors of the adhesively bonded joints for tensile testing, as showed in Table 1. Sandpaper mesh numbers of P80, P180, P320, P600, and P800 were used. The sheets were grinded in a single direction that vertical to the tensile load direction of the adhesive joints for tensile testing, as showed in Fig. 1. The grinding time for each sheet was 10 minutes. In Table 1, sixteen sheets were prepared for each group, and six sheets were used for the test microcosmic surface area, surface free energy and surface roughness respectively, and the remaining ten sheets were bonded into five single lap joints. The surface characteristics were measured at ambient humidity of 42 ± 3% and room temperature of 25 ± 1 °C.

Table 1 The surface treatment of the raw material samples for adhesive bonding

| No | S-0   | A-1  | A-2  | A-3  | A-4  | A-5  |
|----|-------|------|------|------|------|------|
| Sandpaper size | Un-grinded | P80  | P180 | P320 | P600 | P800 |

Fig. 1. The sample size and its measurement points (unit: mm)

2.3 Surface characterization
Two roughness parameters for the substrate are $R_a$ and $R_z$. $R_a$ represents the arithmetic mean of the absolute values of the longitudinal coordinates of the roughness profiles, and $R_z$ represents the arithmetic mean value of the individual roughness depth of successive sampling lengths [24]. $R_z$ was applied to assessment the surface roughness of the samples throughout this study. Surface roughness values, $R_a$ and $R_z$ were measured parallel to the load direction using a profilometer (MarSurf PS 10, Germany). For each sample, 5 points as shown in Fig. 1 were selected for testing, and each point was tested for three times. Three specimens of each parameter group as shown in Table 1 were measured, and the average value was regarded as the ultimate result. The surface morphology characteristics and micro surface area of samples were obtained using a VHX-2000C testing machine.

To accurately obtain the failure pattern and the interface failure area, the failure surface of the adhesively bonded joints was recorded and the failure area was measured using a DXT-45 single-cylinder stereomicroscope. The percentage of interfacial failure is equal to the proportion of interfacial failure area to the whole overlap area in the adhesively bonded joints.

2.4 Contact angle testing and surface free energy calculating

Ordinarily, the considerable level of wettability of aluminum substrate surface is a precondition for good adhesion. It is generally determined by the surface free energy of the substrates to be bonded. The contact angle measurement methods were adopted to compare the change of surface free energy of the aluminum sheets after grinding. The contact angles of testing liquids were performed on a JC-2000D4F analyzer. The
measured values were employed to estimate the surface free energy which was very strongly associated with wettability. The picture of the droplet was captured by a digital camera located in a contact angle measurement instrument, and the images were analyzed using the JC2000DB software. the surface free energies were counted by the Owens-Wendt equation, as shown by Eq. (1). Distilled water and diiodomethane, typical polar and liquid and nonpolar liquid were used, which were selected for contact angles measurement. In the test process, three points were tested on each specimen, and their average values were used as the ultimate results. The test liquid drop volume was 3.36 μl. Surface tension measurement of liquid at room temperature and its component are shown in Table 2. Taking the surface tension of the liquid-solid-gas triangular points into account in equilibrium equation (Eq. (2)), the surface free energy $\gamma_s$ with its dispersive component $\gamma^d_s$ and polar component $\gamma^p_s$ of the substrates were counted [25] as below:

\[
W_a = \gamma_L (1 + \cos \theta) = 2\sqrt{\gamma^p_s \gamma^d_L} + 2\sqrt{\gamma^p_s \gamma^d_L} 
\]

\[
\gamma_s = \gamma^p_s + \gamma^d_s 
\]

where $\theta$ represents the contact angle, and $W_a$ represents the work of adhesion, the total surface energy, $\gamma_s$ represents the sum of the polar component $\gamma^p_s$ and dispersive component $\gamma^d_s$.

For the liquid-solid interaction system through dispersion forces, Eq. (2) can be derived by considering the geometrical average of two liquid dispersion component, and Eq. (3) can be introduced:

\[
\gamma_{sl} = \gamma_s + \gamma_L - 2(\gamma^d_s \gamma^p_L)^{1/2} 
\]

or Eq. (4):
\[ \gamma_{L}(1 + \cos \theta) = 2(\gamma_{d}^{L} \gamma_{p}^{L})^{1/2} \]  

While the diiodomethane and the water contact angles on the samples were tested at the room temperature, the components \( \gamma_{p}^{d} \) and \( \gamma_{d}^{d} \) of the aluminum substrates can be counted according to the Owen-Wendt equations Eqs. (5) and (6):

\[
(\gamma_{S}^{d})^{0.5} = \frac{\gamma_{w} \sqrt{\gamma_{d}^{p}} (1 + \cos \theta_{w}) - \gamma_{d} \sqrt{\gamma_{d}^{p}} (1 + \cos \theta_{d})}{2(\sqrt{\gamma_{w}^{d} \gamma_{d}^{d}} - \sqrt{\gamma_{d}^{d} \gamma_{w}^{p}})} \tag{5}
\]

\[
(\gamma_{S}^{p})^{0.5} = \frac{\gamma_{w} (1 + \cos \theta_{w}) - 2\sqrt{\gamma_{w}^{d} \gamma_{d}^{d}}}{2\sqrt{\gamma_{w}^{p}}} \tag{6}
\]

where: \( \gamma_{d}^{d} \) and \( \gamma_{p}^{d} \) represent the dispersive component and polar component of the surface free energy of the tested samples, respectively; \( \gamma_{d}^{d} \) and \( \gamma_{w}^{d} \) represent the surface free energy of the diiodomethane and water, respectively; \( \gamma_{d}^{d} \) and \( \gamma_{p}^{d} \) represent the dispersive component and polar component of the surface free energy of water, respectively; \( \gamma_{d}^{d} \) and \( \gamma_{p}^{d} \) represent the polar component and dispersive component of the surface free energy of diiodomethane, respectively; \( \theta_{w} \) and \( \theta_{d} \) represent the contact angle of water and diiodomethane, respectively. According to Eq. (2), the surface free energy of joints with different surface treatments can be calculated.

Table 2 The components and its surface free energy of the measured liquids at room temperature, in mN/m.

| Testing liquids | \( \gamma_{L} \) | \( \gamma_{p}^{d} \) | \( \gamma_{d}^{d} \) |
|-----------------|-----------------|-----------------|-----------------|
| Diiodomethane   | 50.8            | 0               | 50.8            |
| Distilled water | 72.8            | 51.0            | 21.8            |

In order to completely comprehend the wetting status of the adhesive on the aluminum surface and to validate the test results for the test liquids, the contact angles of the 1840C structural adhesive on the aluminum substrates surface at 180 °C, which was beyond its melting point, were also tested for comparison. The same testing and
calculating methods as described above were adopted. The only difference was that the
testing platform was constantly heated and kept at the testing temperature.

2.5 Joint preparation and lap shearing strength testing

After surface treating with different sandpaper, the aluminum sheets were cleaned
by acetone with an ultrasonic cleaner for 20 minutes. Then the sheets were dried in the
air at room temperature for 4 hours. Adhesive was uniformly daubed on the aluminum
surface using a hand-held injection gun. In order to control the adhesive thickness, two
copper wires with Φ 0.3mm are inserted into the adhesive layer. The adhesive area was
25 mm in width and 12.5 mm in length. A C-type flat-mouth fixture was used to clamp
the aluminum substrates and adhesive together at a pressure of 0.2 MPa [26]. After
assembly, the redundant adhesive beyond the bonding area was removed. Finally, the
adhesively bonded joints were cured for half a minute in a drying oven at 180 ± 0.2 °C.

Lap shearing test which is a widely used joint test for automobile parts [27], was
conducted to investigate the effect of the different surface roughness of the aluminum
substrates on the shear strength of the adhesively bonded joints. It was performed
through the tensile testing standard after curing. The parameters setting referred to the
standard ASTM D1002-2001. In order to ensure that the force direction is maintained in
a straight line during the test, and to decrease bending stress generated, a shim was
pasted with at the end of each aluminum sheet of the joint. The total shim thickness was
the total of the thickness of the aluminum substrate and the adhesive layer, namely 4.1
mm. Detailed parameters of the adhesively bonded joints for lap shearing strength
testing are shown in Fig. 2. The testing was carried using an Instron 3369 tensile testing
equipment, and the load-displacement curves were acquired, and the loading speed is 5 mm•min\(^{-1}\). The maximum load in load-displacement curve was applied to estimate the lap shearing strength. Tensile tests were performed on three joints for each parameter in Table 1, and the average strength was considered as the ultimate result. Lap shearing strength of the joints was calculated on the basis of Eqs. (7)

\[
\sigma = \frac{F_{\text{MAX}}}{L \times W}
\]

(7)

where \(\sigma\) represents the lap shearing strength of the joints; \(F_{\text{MAX}}\) represents the peak loading force; and \(L = 12.5\) mm represents the length of the bonding area, and \(W = 25\) mm represents the bonding the width of the bonding area. After stretching, the fracture surface of all test joints was analyzed after failure.

Fig. 2. The specimen parameters of aluminum alloy adhesively bonded joints (unit: mm)

3 Results and discussion

3.1 Surface roughness testing results

Fig. 3 indicates the surface roughness measurement results, namely of surface roughness \(R_a\) and \(R_z\) of the aluminum sheets before and after sandpaper grinding. It can be observed that the substrate surface is apparently roughened after grinding. The largest values of \(R_a\) and \(R_z\) of the specimens treated by sandpaper (A-1) are 2.06 \(\mu\)m and 13.74 \(\mu\)m, respectively, which are as high as 7.1 times and 4.8 times that of the un-
grinded simples (S-0), respectively. The $R_a$ and $R_z$ of specimens decrease as the mesh number of sandpaper increases. In general, the smaller the sandpaper mesh number is, the larger the particle size will be. When the sandpaper mesh number turned from P80 to P800, the average variation of $R_a$ and $R_z$ increased 1.54 μm and 9.81 μm, respectively.

![Graphs showing surface roughness Ra and Rz](image)

Fig. 3. Effect of sandpaper grinding parameters on surface roughness (a) Ra and (b) Rz

### 3.2 Lap shearing test results

The lap shearing test results are the most directional reflection of the mechanical behavior of the adhesively bonded joints. Fig. 4 demonstrates the lap shearing strength of the adhesively bonded joints made of the aluminum sheets with different surface roughness values. The maximum profile height $R_z$ is used in this study. As demonstrated in Fig. 4, the lap shearing strength of the joints firstly increases and then slightly decreases with the surface roughness. Compared to the joint made of un-grinded sheets, the lap shearing strength of the joints made of grinded sheets shows a great improvement. It increases rapidly from 19.3 MPa to 30.4 MPa (an increase of 57.5%), when the surface roughness increases from 2.88 μm (un-grinded samples) to 6.08 μm.
(grinded by sandpaper with mesh number of P320). Then with the surface roughness of the aluminum substrate increase, the shearing strength of the joints decreases. When the surface roughness of aluminum is 6.08 μm, the maximum shearing strength of the joint is 30.4 MPa, which increases by 9.7%, compared to the joints with the surface roughness of 3.93 μm, and increases by 57.5%, compared to the joints with the surface roughness of 2.88 μm. In this condition, the lap shearing strength of the joints reaches about 82% of that of the structural adhesive which is about 37.1 MPa. Lap shearing strength decreases when the joints of surface roughness exceed 6.08 μm. For example, the lap shearing strength of the joints were made of the sheets with surface roughness of 13.74 μm is 26.7 Mpa, which is 12.2 % lower than that of the joints made of the sheets whose surface roughness is 6.08 μm.

Fig. 4. Lap shearing strength changes with surface roughness

3.3 The Effect of surface roughening on the lap shearing strength of the joints

3.3.1 Influence of grinding on the surface morphology of the raw materials

The surface roughness affects the lap shearing strength of adhesively bonded aluminum joints through surface morphologies modification. To explore the inherent
mechanism, the surface morphology of the aluminum substrates treated with sandpapers of different mesh numbers were observed and compared. The surface morphology images are presented in Fig. 5. Many parallel valleys are recorded on the surface of the grinded aluminum substrates, and width and depth of these valleys decrease with the increase of the sandpaper number mesh. The surface morphology of the un-grinded sheets is also shown in Fig. 5, in which the color of surface images is lighter compared to the grinded sheets. It means that the un-grinded specimen has a flat surface, and exhibits a small surface roughness value. For the surface characteristics of grinded sample as shown in Fig. 5, the morphology color brightens dramatically and the scratches depth increases with the surface roughness of sheets increases.

As shown by Fig. 4, the picture shows that surface roughness significantly affects the lap shearing strength of the adhesive bonded joints significantly. When the surface roughness of the grinded aluminum substrates is 6.08 μm, the lap shearing strength is 57.5 % higher than that of the joint made of the sheets whose surface roughness is 2.88 μm. The reason is that when the aluminum substrates were grinded by sandpaper, grooves, ridges and other uneven flats were produced on the substrate surface, as shown in Fig. 5(d). It prevents the adhesive from detaching the aluminum substrates. In this way, surface roughening improves the lap shearing strength of joints greatly. The roughening grade also significantly affects the lap shearing strength of the joints. When the aluminum substrate surface roughness made of the sheet is 6.08 μm, the lap shearing strength is 30.4 MPa, which is 9.7 % higher than that of the joints made of the sheet whose surface roughness is 3.93 μm. The reason is that when the aluminum substrates...
were grinded by the coarse sandpaper, so that several hollows were formed on the aluminum substrates surface. The grooves become wider and deeper, and the ridges become wider and higher (illustrated in Fig. 5(b) and (d)). The greater the height difference between the grooves and the ridges is, the more difficult to separate the adhesive from the aluminum substrates will be [23]. It was found that the grinded surface exhibit uniform grooves and ridges surface pattern, as shown in Fig. 5. Grooves and ridges offer more micro surface area for bonding, and increase the opportunities for the adhesive to permeate into the grooves. However, it weakens the interlocking effect of the surface features when the raw sheets were grinded by P800 and un-grinded samples. Therefore, to a certain extent, roughening the aluminum substrate surface enhances the lap shearing strength of the joints.

![Diagram](image_url)
Fig. 5. Surface morphology images of aluminum sheets before and after grinding with different
mesh number of sandpaper (a) Un-grinded; (b) P800; (c) P600, (d) P320, (e) P180, (f) P80

The anchor behavior mechanisms between the substrates and the adhesive indicate
that the grinding surface morphologies have a significant effect on the shearing strength.
As demonstrated in Fig. 6 and Fig. 7, surface roughening obviously increases the
interfacial area ratio. As a result, the number of bonding points at the contact area
between the adhesive and aluminum substrates increases remarkably. In addition, a
large number of anchors allow the adhesive to permeate the surface of the aluminum
substrate, and then the adhesive is interlocked with the substrate in the groove during
curing. Because the grinding process modifies the amount of interaction between the
substrates and adhesive, surface roughening increases the mechanical interlocking effect.

In Fig. 6, the fluctuation of un-grinded samples profile curve is lower than that of the
grinded samples. In the cases, when the surface roughness values are 2.88 μm and 6.08
μm, the mean depth of the grooves aluminum substrates are approximately 0.02 μm and
4 μm, respectively. Moreover, the rougher the aluminum substrate is, the stronger the
fluctuation of the surface profile becomes. When the surface was not grinded, the
surface profile is nearly a straight line. However, as seen from Fig. 6(b) to Fig. 6(f), the
surface profiles are dramatically fluctuant curves when the samples were grinded by
sandpapers. To better clarify the influence of anchoring of adhesively bonded joints, the
cross-section of the bonded samples was examined. Fig. 7 lists the cross-section
morphology of the adhesively bonded joints made of the sheet without grinding sheets
(a), smaller roughness (b), moderate roughness (c), and larger roughness (d), which has
a higher magnification compared to the surface profiles in Fig. 6. It is obvious to see
that the cross-section of Fig. 7(a) shows a nearly straight line, and the cross-section of Fig. 7(d) shows a sawtooth shape. The difference of two types of cross-sections leads to different surface roughness. Combined with Fig. 4, the lap shearing strength of the bonded joints made of grinded sheets is higher than that of joints made of un-grinded sheets. This is related to the stronger anchor effect of the surface morphologies originated from larger contact areas [13]. With larger anchor sizes, it is much more difficult for the adhesive to separate from the aluminum substrates. Analyzing the other samples were grinded by sandpaper, the same effect can be summarized when the surface roughness values are below 6.08 μm.

Fig. 6. The surface roughness profile of aluminum substrates with various surface treatment (a) un-grinded, (b) P800, (c) P600, (d) P320, (e) P180, (f) P80
3.3.2 Influence of grinding on the surface area of the raw materials

Fig. 8 shows the change of microcosmic surface area for grinded sheets compared to un-grinded sheets. It is obviously examined that the microcosmic surface area value of samples grinded by sandpaper is apparently higher than that of un-grinded samples. The microcosmic surface area of un-grinded sheets is $3.2 \times 10^5 \mu m^2$. The higher surface roughness of aluminum substrates is, the larger microcosmic surface area will be. Besides, when the surface roughness is 3.93 $\mu m$ and 13.74 $\mu m$, its microcosmic surface area increment is $3 \times 10^5 \mu m^2$ and $19 \times 10^5 \mu m^2$, respectively. These results indicate that surface roughening can increase the microcosmic area of the aluminum substrate. Furthermore, combined with Fig. 4, it is clear that the lap shearing strength of grinded samples with a rougher surface is greater than that of un-grinded samples when the $R_z$ is between 2.88 $\mu m$ and 6.08 $\mu m$. This is related to that surface roughening improves the interfacial adhesion facilitate the transmission of stress to the substrate.
effective contact areas in grinded samples. Due to the existence of grooves and ridges on the substrate surface, there is more micro surface area for bonding. Therefore, the larger the effective contact area is, the weaker the stress concentration will form, and the adhesively bonded joints are difficult to be damaged \[^{13}\]. Similar trends were observed in average roughness and microcosmic surface area when the $R_z$ is between 2.88 $\mu$m and 13.74 $\mu$m, which proves that the enhanced adhesion caused by mechanical grinding resulting from the increasement of surface area. However, the lap shearing strength of the joint is not a linear relationship with the microcosmic surface area.

![Graph showing the increment of microcosmic surface area for grinded sheets compared to un-grinded sheets.](image)

**Fig. 8.** The increment of microcosmic surface area for grinded sheets compared to un-grinded sheets

### 3.3.3 Influence of grinding on the surface free energy of the raw materials

In order to find out the reasons why the lap shearing strength of the joint does not increase monotonically with the surface roughness increasing, the interfacial adhesion situations of the joints with grinded aluminum substrates surface were investigated. Wettability is an important index to evaluate interfacial adhesion situations, which can help engineers to comprehend the interaction mechanism between the adhesive and substrate. Wetting behavior of the adhesive on the aluminum sheets with different
surface morphology, and roughness was analyzed based on the method reported in the literature [28]. Fig. 9 shows the average water contact angles and diiodomethane contact angles on the aluminum substrate surface after grinding with the different number mesh sandpaper, respectively. As the surface roughness of aluminum substrate increases, the surface contact angle decreases firstly and then increases gradually in the test liquid using both water and diiodomethane. Furthermore, when the surface roughness of the aluminum substrate is 2.88 μm, the water contact angle is 67.21°, and then it decreases dramatically when the surface roughness turns from 2.88 μm to 6.08 μm, and further drops to 42.25° when the surface roughness increases to 6.08 μm. After that, when the surface roughness is 13.74 μm, the water contact angle grows quickly to about 53.94°. The changing trend of the diiodomethane contact angle is the same as that of water. When the surface roughness of the aluminum substrate is 2.88 μm, the diiodomethane contact angle is 47.18°, it drops to 27.97° when the surface roughness increases from 2.88 μm to 6.08 μm. After that, the diiodomethane contact angle grows quickly to about 35.78° when the surface roughness of the aluminum substrate reaches to 13.74 μm. The water contact angles and diiodomethane contact angles on the grinded aluminum substrate are significantly lower than that of un-grinded samples. According to Eq. (1), the smaller the contact angle of the aluminum substrate means it has higher $W_a$ and better wettability. The wettability firstly becomes better and then goes worse with the surface roughness increasing. Surface wettability of the samples is deeply affected by the surface roughness. When surface roughness exceeds 6.08 μm, the wettability and
interfacial bonding effect become poor, which results in the lap shearing strength decrease.

![Graph](image)

Fig. 9. The contact angle of testing liquid with different surface roughness (a) water (b) diiodomethane

The contact angles of the 1840C structural adhesive at 180 °C on the aluminum substrate with different surface roughness values are shown in Fig. 10. It can be found that the changing trend of adhesive contact angle is consistent with that of water and diiodomethane. When the surface roughness increases from 2.88 μm to 6.08 μm, the adhesive contact angle decreases from 66.2° to 55.4°. Meanwhile, the wettability of the aluminum substrates increases with the surface roughness increasing. The fact is that the interfacial area both in the substrate and adhesive is largely increased. When the surface roughness of the substrate reaches to 6.08 μm, the aluminum substrate has the best wettability. The infiltration of the adhesive on a metal substrate was affected by many factors, for example, the geometry of the dimples, the surface free energy and the rheology of the adhesive. For the adhesively bonded aluminum joints, even if the oils and organic contaminants of the substrate surface are removed, the spreading of adhesive on the aluminum substrates surface is difficult because the surface free energy
of the substrates is very low. Therefore, the adhesive cannot infiltrate every corner of
the aluminum substrate surface.

Fig. 10. The adhesive contact angle with different surface roughness at 180 °C

According to the absorption theory, the surface and interfacial energies are related
to the interfacial strength between the substrate and the adhesive, which influences the
strength of the adhesively bonded joints [29, 30]. Fig. 11 shows the influence of the
surface roughness on the surface free energy and its component calculated according to
Eq. (1) and Eq. (2). From these results, it seems that the surface free energy of the
aluminum substrate has the opposite change trend compared to the surface contact angle
with the increase of surface roughness. However, the surface free energy of aluminum
substrates and the lap shearing strength of the adhesively bonded joints have the same
change trend with the surface roughness increase. The dispersion component
(diiodomethane) and polar component (water) of surface free energy of various samples
are apparently different from each other. The polar components (water) surface free
energy is generally lower than that of the dispersion components (diiodomethane).

Although there is no obvious relationship between the surface free energy and surface
roughness, the surface free energy of the dispersion component (water) is much closer
to the total surface free energy. One reason is that polar elector-donor interactions
decrease with the surface roughness and the nonpolar dispersion interactions do not rely
on the surface morphology. The adhesive adsorption theory holds the opinion that
bonding is the use of mechanical bonding force, chemical bonding force and physical
adsorption force between the interfacial connecting materials. Because the electronic
polarity of the adhesive and aluminum substrate attracts each other to produce a
bonding force, the adhesively bonded joints become stronger, which leads to less
interfacial failure area. As a result, the failure percentage of the interfacial contact area
initially decreases and then increases with the surface roughness, as shown in Fig. 6.
Therefore, in view of the adhesive adsorption theory, the changing trend of interfacial
failure percentage is opposite to that of the lap shearing strength with the surface
roughness. Similarly, the lap shearing strength of the adhesively bonded joints firstly
increases and then gradually decrease as the surface roughness increases according to
the theory. Generally, with the increase of surface roughness of the sheets, the surface
free energy calculated by the water contact angle decreases. Moreover, the polar
component of the surface free energy increases from 35.83 mJ/m² to 45.03 mJ/m² when
the surface roughness of the aluminum substrate increases from 2.88 μm to 6.08 μm. In
all cases, the aluminum substrate surface was grinded by the different number mesh of
sandpaper, and the percentage of the polar component of surface free energy is greater
than that of the dispersive component. These results are consistent with the previous
studies [24, 31]. The surface free energy values of the sample with surface roughness of
2.88 μm is 45.89 mJ/m² which are 14.8% and 29.5% lower than those when the surface
roughness is 3.93 μm and 6.08 μm, respectively. Meanwhile, the surface free energy of
the sample with a surface roughness of 13.74 μm is 56.69 mJ/m², which is 12.9% lower
than that of the sample when the surface roughness is 6.08 μm. Thus, when the surface
roughness of aluminum sheets is 6.08 μm, it has the highest surface free energy. As
pointed out by Harris and Beevers [32], smoother sandblasting surfaces have higher
surface free energy. Furthermore, Hitchcock et al. [33] examined that the surface
roughness of the substrate usually leads to the decrease of wettability in a certain range,
too. Some researchers found out that the asperities, ridges and peaks, on the surface of
the substrates produce obstacle to prevent droplet diffusion [34, 35]. Thus, over
roughening surface greatly decreases the surface free energy of the aluminum
substrates, and reduce the lap shearing strength of adhesively bonded joints.

According to Eq. (3) and (4), the adhesion energy of adhesive/aluminum substrate
is calculated by combining the surface free energy of the adhesive and the test contact
angle. The results are shown in Table 3 and Fig. 11. The simplified zero diffusion
pressure is the premise for calculating the adhesion work in ambient air using Eq. (3).
Because the work of adhesion, \(W_a\), determines the status that adhesive penetrating into
the substrate, the bonding work has a significant impact on the diffusion of the adhesive.
When the bonding joints are heated to 180 ° C, this temperature is also the curing
temperature of the adhesive. The adhesive will form a specific contact angle on the
surface of the aluminum plate to wet the aluminum substrate. The contact angle of the
adhesive on the aluminum substrate is 55.4° ± 0.8°. According to the reference [36], the
surface free energy of structural adhesive at room temperature is 65.06 mJ/m² (\(\gamma_s = \gamma_f + \)
\( \gamma_d = 20.03 \text{ mJ/m}^2 + 45.03 \text{ mJ/m}^2 \). Then, according to the test value of contact angle (55.4°) and the surface free energy of the structural adhesive (65.06 mJ/m\(^2\)), and the adhesion work between aluminum substrate and adhesive is 102.14 mJ/m\(^2\) calculated by Eq. (1).

The work adhesion increases with increasing surface roughness in the range from 2.88 \( \mu \text{m} \) to 6.08 \( \mu \text{m} \). One factor improving the lap shearing strength is that the mechanical interlocking effect of the metal substrates. In order to achieve a good mechanical interlock effect between the adhesive and the substrate, a perfect infiltrating of the adhesive into the irregular corners or the grooves on the substrate surface is necessary. Consequently, surface roughening produces adhesive interfacial adsorption that can improve the mechanical interlock effect. Another significant factor which promotes the bonding strength is that a satisfied adhesion work is needed, although its value is quite lower than that of the actual separation work. Because of the tested value of the adhesion work \( W_a = 102.14 \text{ mJ/m}^2 \) in the adhesive/aluminum bonding system is higher than 65.06 mJ/m\(^2\), it is sufficient to produce a mechanical interlocking. Under this condition, the adhesive can completely flow into all corners of the micro roughened aluminum substrate.

| Table. 3 Surface free energy and its components for substrates with different surface roughness |
|---------------|-------|-------|-------|-------|-------|-------|
| \( R_z (\mu \text{m}) \) | 2.88  | 3.93  | 4.69  | 6.08  | 7.84  | 13.74 |
| \( \gamma_p \) (mJ/m\(^2\)) | 35.83 | 41.74 | 43.95 | 45.03 | 42.71 | 41.66 |
| \( \gamma_d \) (mJ/m\(^2\)) | 10.06 | 12.14 | 17.86 | 20.03 | 15.53 | 15.03 |
| SFE (mJ/m\(^2\)) | 45.89 | 53.88 | 61.81 | 65.06 | 58.24 | 56.69 |

*Note: SFE is short for surface free energy.*
3.4 Failure behavior of the adhesively bonded joints

The failure mode of adhesively bonded joints after the tensile test usually includes three categories, namely the cohesive failure, interfacial failure, and mixed failure modes [37]. Cohesion failure is a fracture of the adhesive itself. The interfacial failure mode is defined as the adhesive is completely separated from the substrate surface. However, the mixed failure mode refers to some cohesive failure and some interface failure. In practical application, most of them are mixed failure mode. In this way, the engineers try to make the most of the mechanical performance of the structural adhesive. The failure patterns of adhesively bonded joints after the test are shown in Fig. 12. The joint made of un-grinded sheets have the biggest interfacial failure area among all the different treatment parameters joints after lap shearing tests. Moreover, the interface failure area initially decreases and then increases with the surface roughness increases. When the surface roughness of aluminum sheet is 6.08 μm, the interface failure area rarely exists in the failure pattern. Compare to the failure pattern of
other treatment parameters joints, the number of bare regions in the fractured surface is the least.

Fig. 12. The macro-morphology of fractured surface with different mesh number of sandpaper (a) un-grinded; (b) P800; (c) P600; (d) P320; (e) P180; and (f) P80.

The interface failure area percentages of the adhesively bonded joints is demonstrated in Fig. 13. With the surface roughness of the aluminum substrates increases, the percentage of interface failure area of adhesively bonded joints initially decreases drastically and then increases slightly. The percentage of interface failure area of the joint made by the substrates with surface roughness of 9.8 μm drops by 76.4% approximately, compared to the adhesively bonded joints made of un-grinded substrates. It indicates that the surface roughening reduces the percentage of interface failure significantly and enhances the lap shearing strength. In addition, the smaller the interface failure percentage is, the stronger the adhesively bonded joints strength will be. When the surface roughness is 6.08 μm, the adhesively bonded joints have the
smallest interface failure area, which decreases by 92.5% and 68.3% for those bonded joints with the surface roughness of 2.88 and 13.74 μm, respectively.

Fig. 13. The bare area on the fracture surface

3.5 Influence of over roughening on the failure behavior of the joints

As illustrated in Fig. 4, the lap shearing strength decreases apparently when the surface roughness turns from 6.08 μm to 13.74 μm. In another word, over roughening surface of raw materials is harmful to the interfacial adhesion state of joints cross-section. Fig. 14 depicts the OM images of the adhesively bonded joints cross-section with coarse surface treatments (grinded by P80 and P180). It can be seen that over roughening produces micro-cracks at the interfacial region, which is consistent with the previous studies [23, 38]. These defects will lower the effective contact area so that lap shearing strength decreases. Moreover, the extremely uneven surface prohibits the adhesive in freely spreading on the substrate surface. Based on the information mentioned above, taking adhesively bonded joints as examples, Fig. 15 depicts a mechanism diagrammatic of the cross-section of the joints with different surface roughness. Fig. 15(a) shows the mechanism diagram of the bonding status of the coarse
interface, indicating the samples grinded by sandpaper with the P80 and P180, whose surface roughness is greater than that of the other three treatment methods and un-grinded, but the bonding status is extremely bad, meaning their mechanical interlocking effect is very terrible. Some reasons are concluded as below: (i) the profiles were disorder and nonuniform on the micro-scale aluminum substrate surface, as shown in Fig. 6. It will cause the adhesive to be irregularly distributed on the aluminum substrate surface. (ii) Because air gas is trapped in the valleys, over roughening substrate surface prevents the adhesive from wetting the coarse surface of the aluminum substrate completely. (iii) Because of insufficient wettability and residual air between the aluminum substrate and the adhesive, the corners and grooves on the substrate surface cannot entirely saturated with adhesive before solidification. Those factors reduce the valid bond area and create stress concentration at the interfacial region. As a result, when the aluminum substrate surface is grinded by P80 or P180, the surface is coarse, and a lot of underfill areas of the joints will be formed, and an alternating solid-liquid and gas-liquid interface will also gradually appear as shown in Fig. 14 and Fig. 15(a).

Fig. 14. OM images of the adhesively bonded joints cross-section with different surface treatment (a) grinded by P180, (b) grinded by P80
Fig. 15. The interface bonding diagram of different surface treatment prepared with (a) coarse; (b) moderate; (c) fine

Fig. 16. Failure pattern (a) grinded by P180; (b) grinded by P80

It can be seen from Fig. 15 that the penetration of the epoxy on the grinded aluminum surface has two different modes. Fig. 15 (a) illustrates the distribution of the epoxy on the coarse substrate with surface roughness, for example the sheets grinded by P80 and P180, while Fig. 15 (b) and (c) depicts the distribution of epoxy on the sheet with moderate and fine roughness, representing the cross-section of adhesive joints made of sheets which were grinded by P320, P600 and P800 and un-grinded sheets.
surface, respectively. The capillary wettability mechanism of the grooves is applied to explain the observed penetration process of epoxy [35]. The adhesive flows into the grooves driven by the capillary force. The capillary wettability of the uneven surface can be influenced by two key factors: the percentage of the groove depth to groove diameter and the percentage of the groove diameter to the groove space. When the percentages are at appreciate values, the substrates have the excellent wetting. Moreover, the epoxy wetting mechanism changes with the different surface microstructure of aluminum substrates. To verify the hypothesis, it is obvious to see that many voids in the failure pattern of adhesively bonded joints after the shearing test as shown in Fig. 16. It means that the adhesive is not entirely contacted with the aluminum sheets. The lap shearing strength of adhesive joints with over roughening surfaces is very low because of many internal voids and other defects in the joints.

4 Conclusions

In this research, the influence of surface roughness on the lap shearing strength and the failure behavior of the adhesively bonded aluminum joints were experimentally studied. The related mechanisms were systematically investigated. Main conclusions can be drawn as follows:

(1) The lap shearing strength of the adhesively bonded joints was obviously influenced by the surface roughness of the aluminum substrate. With the surface roughness of the aluminum substrate increased, the lap shearing strength of the joints initially increased and then decreased. When the surface roughness of the aluminum
substrate reached 6.08 μm after roughening with sandpaper, a maximum lap shearing strength of 30.4 MPa was obtained for the joint, improved by 57.5% compared with that of the joint made by sheets with surface roughness of 2.88 μm without roughening. Beyond that, the lap shearing strength decreases with the roughness of the substrates decreasing.

(2) The mechanism for the change of the lap shearing strength with the surface roughness is related to the modification of the mechanical interlocking effect, and the microcosmic area, and wettability of the adhesive on the substrate. When the surface roughness of the aluminum substrate ranged from 2.88 μm to 6.08 μm, the lap shearing strength of the adhesively bonded joints was mainly increased by increased microcosmic surface area and improved mechanical interlocking. When it is in the range of 6.08 μm to 13.74 μm, the strength of the joints was mainly affected by the wettability by which the surface energy is obviously decreased. Over roughening would worsen the wettability of the aluminum substrate which leads to a weakened lap shearing strength of joints.

(3) The failure behavior of the adhesively bonded aluminum joint was to a large extent by the surface roughened of aluminum sheets. The relation between the interfacial failure proportions to the surface roughness follows the same changing trend of the strength to the surface roughness, which also decreases firstly and then increases. Proper surface roughening can increase the micro surface area, which can reinforce the lap shearing strength. However, over roughening would induce voids, which turns the failure mode from the mixed mode of adhesion failure and interfacial failure to the
single interfacial failure, by which the baring ability of the interface is largely
decreased.

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