Effect of ultrasonic vibration on adhesive enhancement of plasma-modified nickel surface

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1. Introduction

Carbon fibre-reinforced polymer (CFRP) has been widely used in construction [1,2], automobile [3,4], aerospace [5,6] and other fields owing to its light weight, high strength, corrosion resistance and strong plasticity. For a helicopter rotor blade, its beam, skin and other structures, which are mainly load-bearing components, have gradually changed from metal materials such as stainless steel and aluminium alloy to CFRP and glass fibre-reinforced polymer (GFRP) [7,8]. In order to protect the composite blade and allow it to resist dust, lightning, icing and other environments, a metal shield needs to be installed at the leading edge [9]. The shield is usually made of nickel, stainless steel or titanium alloy. Unlike traditional metal materials, the composite materials cannot be connected by bolting or riveting without damaging the performance of the materials, for puncturing inevitably cuts off some fibres and breaks the materials. Therefore, the composite materials and metal materials are mainly connected by adhesive bonding [10].

The rotor blade skin of many aircraft is made of CFRP, and the leading edge of the blade is bonded with a nickel shield [11]. However, some shields were accidentally debonded during service [12]. By examining a debonded shield, it was found that most of the adhesive was bonded to the surface of the composite blade, while there was almost no residual adhesive on the surface of the nickel shield. It indicates that the bonding performance of the nickel surface is far lower than that of the CFRP. Accordingly, it is necessary to properly modify the nickel surface to improve the adhesion of CFRP/Ni.

Various surface modification methods are developed for metal adherends, including physical methods such as grinding and sandblasting, and chemical methods such as anodizing, coupling treatment and plasma treatment [13–16]. The plasma technology is widely used in surface modification of wood, composite materials and metals because of its high efficiency, environmental protection and nondestructive nature [17]. A plasma is a gas in which quite a lot of atoms or molecules are ionized under an applied electric field, and it belongs to the fourth state

Keywords: Ultra sonic vibration Plasma modification Sandblasting Adhesive bonding Strengthening

Abstract

Poor adhesion of nickel surface limits its further application in the aerospace field. In this study, plasma modification was conducted on the surface of the nickel plate pretreated by sandblasting, and then ultrasonic vibration was applied during the adhesively bonding process of the CFRP(Carbon fibre-reinforced polymer)/Ni joints. The bonding strength of the joints was increased by 65%. The adherend surface and the bonding interface were analyzed from microstructure, element distribution and chemical bonding to study the strengthening mechanism. By the sandblasting, irregular pits were formed on the nickel surface, effectively increasing the surface roughness. The plasma modification could introduce active functional groups including hydroxyl, amino and carboxyl on the nickel surface, which improved the surface wettability macroscopically. However, at a microscopic level, the adhesive with high viscosity and poor fluidity did not form a compact interface with the nickel. The ultrasonic application could promote the filling of the adhesive in irregular micro-scale pits on the surface, thereby strengthening the mechanical anchoring effect. Furthermore, the ultrasonic application produced dynamic impingement at the interface, enhancing the contact between the adhesive and the nickel plate. The adhesive molecules could fully collide and react with the active functional groups introduced on the nickel surface to form more chemical bonds, thus effectively improving the bonding strength of the CFRP/Ni joints.
of matter. Plasma surface modification can directly or indirectly introduce active functional groups onto the surface of a material to improve the wettability, biocompatibility and electrical properties [18]. Miyau et al. [19] investigated the influence of a low-pressure O₂/Ar gas plasma modification of a polyimide film on the adhesion to an alloy plating layer made of nickel (Ni) and chromium (Cr). The results showed that the changes in the roughness and an increase in the percentage of hydrophilic groups, particularly C–O and C—OH bonds, improved the adhesive strength between the metal and polyimide. Song et al. [20] treated the surface of polycarbonate (PC) with chemical agents and DBD (dielectric barrier discharge) plasma to improve the adhesion of electrolyte-free nickel plating on the PC. The results showed that the wettability of the PC surface increased with the etching and plasma treatment time, and the adhesion with the DBD plasma treatment was better than that with the chemical treatment. Vivet et al. [21] studied the surface properties of Ni films subjected to an Ar-H₂ RF (radio frequency) plasma treatment and optimized the parameters through experiments. The results showed that the plasma treatment changed the hydrophobic surface to a strong hydrophilic one, and thereby facilitated the electronic and mechatronic assembly process.

A plasma treatment has little etching effect and is generally not applied to metal surfaces alone. In an actual bonding process, a metal surface is often pretreated by physical methods, such as grinding or sandblasting, to increase the surface roughness. However, adhesive always with high viscosity and poor fluidity might not make good contact with the roughened surface. As a result, active functional groups introduced by a plasma treatment cannot be fully utilized to form chemical bonds with adhesive, which is not conducive to the bonding.

In recent years, ultrasonic vibration has been widely used in promoting filling and wetting of adhesive [22,23]. Liu et al. [24] investigated the effects of ultrasound on the surface tension of the epoxy resin and on the surface characteristics of the aramid fibre. The wettability between the aramid fibre and epoxy resin was improved by using ultrasound with a frequency of 20 kHz during the winding process of the composite, so that the interfacial shear strength of the aramid/epoxy composite was 26% higher than that of the untreated one. Yan et al. [25] developed a mathematical model to determine the adhesive penetration ability based on the fluid mechanics and capillary rise theory, and studied the effect of ultrasonic vibration on adhesive bonding performances of ceramic matrix composites. The experimental results showed that the ultrasonic vibration-assisted process could improve the bonding strength. Holtmannspötter et al. [26,27] presented an approach to improve interface formation and ensure the quality of adhesive bonding by using acoustic cavitation in liquid adhesive. Our previous studies have proved that ultrasonic vibration can promote the penetration of adhesive on the surface of adherends, enhance mechanical anchoring effect and promote chemical reaction at the interface [28,29]. However, there is no relevant research on the effect of ultrasonic vibration on adhesive bonding of plasma-modified surfaces.

In this study, surfaces of nickel plates were pretreated by sandblasting and then modified by plasma. The effect of the plasma treatment on the bonding strength of CFRP/Ni joints was analyzed. Then ultrasonic vibration was applied to the bonding process, and the effect of ultrasonic vibration on the bonding was studied. The nickel plate surface and the Ni/adhesive interface were analyzed from microstructure, element composition and chemical reaction to explain the strengthening mechanism.

The rest of this paper is organized as follows. Section 2 describes the experimental materials, processing methods and characterizations. In Section 3, the experimental results are given and analyzed. Finally, conclusions are drawn in Section 4.

2. Materials and methods

2.1. Materials

The bonding plates used are T300 CFRP laminates (Shenzhen Liuliushun Carbon Products Co., Ltd., Shenzhen, China) and N6 pure nickel plates (Xingtai Bodun Cemented Carbide Co., Ltd., Xingtai, China). With T300-3 k carbon fibre as the reinforcement and bisphenol A epoxy resin as the matrix, the CFRP laminates were formed by hot pressing at 150 °C and 30 MPa for 3 h. The thickness tolerance of the laminates is ±0.05 mm, and the interlayer shear strength is 80 ± 5 MPa. The tensile strength of the nickel plates is about 539 MPa, and the chemical composition is listed in Table 1.

The CFRP laminates and the nickel plates were bonded with a toughened dual-component adhesive LOCTITE EA 9309.3NA. The component A of the adhesive is a pink viscous liquid, mainly containing bisphenol A epoxy resin, and the component B is a blue liquid, mainly containing 3,3′-[Oxybis(2,1-ethanediyl)oxy]bis-1-propanamine. The components A and B should be thoroughly mixed at room temperature with a mass ratio of 100:22. The mixed adhesive should be used within the pot life of 35 min and cured for 3 ~ 5 days at room temperature or 1 h at 82 °C to achieve normal performance.

2.2. Methods

Owing to poor bonding performance of the nickel surface, CFRP/Ni joints always debonded at the interface of the adhesive and nickel if no significant modification was applied to the nickel surface. Accordingly, this study mainly focused on surface modification of the nickel plates to improve their bonding performance. The bonding surface of the CFRP laminates was sanded with 400-mesh sandpaper as usual.

2.2.1. Plasma surface modification

The plasma treatment was performed by CRF AP plasma processor produced by Wenzhou Yihong Technology Co., Ltd., as shown in Fig. 1, and its related parameters are listed in Table 2.

The plasma treatment process of a nickel plate is shown in Fig. 2. First, the plate was cleaned with acetone, and then it was placed into deionized water at 60 °C and ultrasonically cleaned for 5 min. The cleaner was JP-010T provided by Skymen Cleaning Equipment Shenzhen Co., Ltd. The ultrasonic frequency used was 40 kHz, the ultrasonic power was 80 W, and the water volume was 1.5 L. Afterwards, the surface of the plate was roughened. The commonly used methods include grinding and sandblasting. In this paper, the sandblasting method was selected because of its better consistency. The sandblasting machine used was WLX-1010 produced by Wuhan Weilixin Machinery Equipment Co., Ltd. Based on results obtained from the process optimization of the sandblasting experiments, the bonding surface of the nickel plate was vertically sandblasted with 80-mesh carborundum under a pressure of 0.5 MPa to remove the surface oxide layer and increase the surface roughness. The sandblasting distance was 50 mm, and the speed was 5 mm/s. After the sandblasting, the nickel plate was ultrasonically cleaned with deionized water and then dried in a vacuum oven at 60 °C. Finally, an air plasma treatment was performed on the surface of the nickel plate. The treatment parameters, including the processing distance, time and power, were optimized by orthogonal experiments, and the experimental results are presented in Section 3.1. After the plasma treatment, the nickel plate was cooled at room temperature, and then was used to conduct the adhesive bonding within 2 h.

2.2.2. Adhesive bonding

After surface modification, the CFRP laminates and nickel plates were bonded using the epoxy adhesive. The joints were designed according to the standard of GB/T 7124–2008, as shown in Fig. 3. The size of the adherends is 100 × 25 × 1.6 mm, and that of the bond line is 25 × 12.5 × 0.2 mm. The clamping area is 25 × 37.5 mm.
Table 1
Chemical composition of N6 pure nickel.

| Element | Ni  | Cu  | Si  | Mn  | C   | Mg  | Fe  | S   |
|---------|-----|-----|-----|-----|-----|-----|-----|-----|
| Mass fraction/% | ≥99.5 | ≤0.10 | ≤0.10 | ≤0.05 | ≤0.10 | ≤0.10 | ≤0.10 | ≤0.005 |

In order to ensure the thickness of the bond line and improve the bonding stability, a fixture made of 7075 aluminium alloy was used to bond the CFRP laminates and the nickel plates. The schematic diagram of the bonding process is shown in Fig. 4. A CFRP laminate was placed on the lower cavity of the fixture, while a nickel plate was placed on the upper one. The height difference between the two cavities is 1.9 ± 0.02 mm. The nominal thickness of the CFRP laminate is 1.6 ± 0.05 mm. Hence, a compensation in thickness was needed to achieve the bond line thickness of 0.2 mm after the CFRP laminate was put into the lower cavity of the fixture, while a nickel plate was placed on the upper one. The height difference between the two cavities is 1.9 ± 0.02 mm. According to $T_c = H_f - T_l - 0.2$ (mm), steel strips (0.01 mm and 0.05 mm in thickness) were placed under the CFRP laminate to obtain the bond line of 0.2 mm in thickness. Together with the fixture, the joint was kept in an oven at 82 °C for 2 h to cure the adhesive completely. After curing, the joint was cooled to room temperature naturally.

2.2.3. Ultrasonic Vibration-Assisted bonding

Ultrasonic vibration was applied during the bonding using the ME-1800 ultrasonic-vibration equipment produced by MAXWIDE ULTRASONIC Co., Ltd., as shown in Fig. 5(a). The ultrasonic equipment comprises an ultrasonic generator, an ultrasonic transducer, a horn and a sonotrode. The ultrasonic generator converts the normal electricity into high-frequency electrical signal matched with the transducer. The transducer receives the electrical signal and converts it into ultrasonic vibration, which is then amplified by the horn and transmitted to the sonotrode. Different ultrasonic frequencies, amplitudes and durations can be achieved by adjusting the ultrasonic generator. The ultrasonic system was fitted with a pneumatic mechanism by which the sonotrode can move up and down.

The joint was fixed on the platform, as shown in Fig. 5. After the process parameters were set, the ultrasonic sonotrode was pressed onto the CFRP laminate, and then the ultrasonic generator was turn on to exert ultrasonic vibration. The ultrasonic vibration was transmitted to the adhesive layer through the CFRP laminate, which affected the bonding process. According to the optimal process parameters obtained from our previous study [30], which applied a similar bonding joint both in size and material, the vibration frequency was 25 kHz, the amplitude was 24 μm, the preload was 0.4 MPa, and the vibration position was 30 mm away from the nickel plate. In order to prevent the CFRP laminate from overheating and ablation, a pulse mode of vibration was adopted. The vibration was kept for 2 s in each cycle of 3 s, and the total time was 48 s. After the ultrasonic vibration was completed, the joint was put into an oven for curing.

2.2.4. Experimental scheme

In order to analyze the influence of the different surface treatments and the ultrasonic application on the bonding performance, four groups of experiments shown in Table 3 were designed. Group A was a reference experiment in which the surface of the nickel plates was only cleaned with acetone. In Group B, the sandblasting treatment was added on the basis of Group A. Based on Group B, the plasma treatment was added in Group C, that is, the plasma modification was performed on the bonding surface of the nickel plates after the sandblasting. The joints in Group A, B, and C were adhesively bonded without ultrasonic vibration. The surface treatment of Group D was the same as that of Group C, but the ultrasonic vibration was applied in the bonding process.

2.3. Characterisation

2.3.1. Bonding strength test

In this study, the tensile shear strength was used to evaluate the bonding strength of the CFRP/Ni joints. The tensile tests were performed on a universal tensile testing machine (SANS CMTS205 manufactured by MT5 SYSTEMS (CHINA) Co., Ltd.), as shown in Fig. 6. According to the standard of GB/T 7124–2008, two heel blocks of 1.8 mm thickness were placed at the two ends of a sample to eliminate the influence of the bending moment on the test results. The tensile tests were conducted at a constant tensile speed of 4 mm/min and the failure time was between 65 ± 20 s. The maximum load was recorded as the...
failure load, and the shear strength was calculated by the following formula:

\[ \tau = \frac{F}{S} \]  

(1)

where \( F \) represents the failure load of the bonding joint (N), \( S \) represents the bonding area (mm\(^2\)), and \( \tau \) represents the shear strength (MPa). The average bonding strength of the five samples in each group was calculated.

2.3.2. Morphology of surface and interface
The surface of the nickel plates and the interface of Ni/adhesive were observed by TESCAN MIRA4 scanning electron microscope (SEM). The nickel plates were treated according to Table 3, and then samples of 10 \( \times \) 10 \( \times \) 1.6 mm in size were cut from the position shown in Fig. 7(a). The samples were observed by the SEM after ultrasonic cleaning and gold coating.
coating to analyze the influence of the different treatments on the surface morphology.

The joints in Group C and Group D were cut into samples of $10 \times 10 \times 3.4$ mm according to the position shown in Fig. 7(b). Then 1000-mesh, 1500-mesh and 2000-mesh sandpapers were used to sand the cross-section of the samples sequentially. After cleaning and coating, the samples were observed by the SEM to analyze the effect of the ultrasonic vibration on the bonding interface.

The morphology and roughness of the nickel surface were tested with Nanoscope IV atomic force microscope (AFM). Samples shown in Fig. 7(a) were taken for the AFM test to analyze the surface morphology and surface roughness of the nickel plates with the different surface treatments.

2.3.3. Chemical characterization of surface and interface

The surface of the nickel plates with the different surface treatments

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**Table 3**

| Group | Sample Number | Surface Treatment Method | Bonding Process |
|-------|---------------|--------------------------|-----------------|
| A     | 1, 2, 3, 4, 5 | Cleaning                 | Traditional bonding |
| B     | 1, 2, 3, 4, 5 | Cleaning & Sandblasting  | Traditional bonding |
| C     | 1, 2, 3, 4, 5 | Cleaning & Sandblasting & Plasma treating | Traditional bonding |
| D     | 1, 2, 3, 4, 5 | Cleaning & Sandblasting & Plasma treating | Bonding with ultrasonic vibration |
was scanned by Xplore 30 energy dispersive spectrometer (EDS), and the element types and contents were analyzed. The scanning position was the same as that observed by the SEM. The functional groups on the surface and the bonding interface were tested using a smart Fourier transform infrared spectrometer (FTIR) produced by Thermo Nicolet corporation. Powder on the surface of the nickel plates was scraped for the FTIR test to analyze the effect of the different surface treatments on the surface functional groups. Powder at the bonding interface was scraped for the FTIR test to analyze the effect of the ultrasonic action on chemical bonding at the Ni/adhesive interface.

3. Results and discussion

3.1. Orthogonal optimization

The orthogonal experimental design is a useful method for studying multi-factor and multi-level experiments. In this study, orthogonal experiments were designed to optimize plasma treatment parameters. The parameters mainly include the processing distance, the processing time and the processing power. According to the specification of the plasma processor, discharge instability may occur when the processing power is lower than 200 W. Generally, the practical processing power is 200 ~ 500 W, and the processing distance is 5 ~ 20 mm. The processing time depends on the sample type and area. According to preliminary trials, the processing time was set as 20 ~ 80 s. The designed factor levels of the orthogonal experiments are listed in Table 4.

Based on Table 4, an orthogonal experimental scheme was designed as shown in Table 5 using the Minitab software (V17), and each case was repeated five times. The joints were bonded without an ultrasonic treatment. The thickness of the bonded joints was measured, and the results showed that the thickness of the bond line was 0.2 ± 0.02 mm, 

![Fig. 6. Tensile test.](image)

![Fig. 7. Observation position for morphology of (a) the surface and (b) the interface.](image)

| Table 4 | Factor levels of orthogonal experiments. |
|---------|------------------------------------------|
| Levels  | Factors       | Processing Distance (mm) | Processing Time (s) | Processing Power (W) |
| 1       | 5            | 20                        | 200                 |
| 2       | 10           | 40                        | 300                 |
| 3       | 15           | 60                        | 400                 |
| 4       | 20           | 80                        | 500                 |
bonding strength. The small error margins reflected a high confidence level of the experimental survey. The results were then analyzed using the “Analyze Taguchi Design” function of the Minitab software. The mean response is given in Table 6, and the main effect plot is shown in Fig. 9.

From Table 6, it can be seen that the influence rank of the parameters is the processing time > the processing distance > the processing power. Fig. 9 shows variation of the bonding strength with factor levels. It can be concluded that the processing distance of 5 mm, the processing time of 60 s, and the processing power of 500 W are the optimal parameters for the plasma treatment. Since the optimal case was not included in the orthogonal experimental scheme, supplementary experiments were required for the verification. The experimental results using the optimal parameters are shown in Fig. 10 (Group C). The bonding strength was slightly higher than the largest one in Table 5 (No.4), which proves the effectiveness of the optimization. The optimal parameters were used for the following study.

3.2. Bonding strength and failure mode analysis

The tensile test results of the four groups of different treatments are shown in Fig. 10.

The average bonding strength of the joints of Group A was 16.3 MPa. After the sandblasting treatment of the nickel plates, the average bonding strength of Group B increased to 19.5 MPa, which was 19.6 % higher than that of Group A. After the sandblasting and plasma treatment on the surface of the nickel plates, the average bonding strength of Group C was 20.6 MPa, which was only 5.6 % higher than that of Group B. It was inconsistent with the expected effect of the plasma treatment on improving the bonding strength. The surface treatment in Group D was the same as that in Group C, but the average bonding strength was significantly increased to 26.9 MPa with the ultrasonic vibration. It was 30.6 % higher than that of Group C and 65 % higher than Group A, indicating that the ultrasonic action could further improve the bonding strength of the CFRP/Ni joints.

The samples after the tensile test are shown in Fig. 11. The failure modes were analyzed according to the standard ASTM D5573-99. For the samples in Group A, all adhesive was observed on the surface of the CFRP laminates, and the failure occurred at the Ni/adhesive interface. Therefore, the failure mode of Group A was the adhesive failure, indicating that the bonding performance of a pristine nickel surface was poor. After the sandblasting, some samples in Group B showed a mixed failure mode of the adhesive failure and fibre-tear failure, while others in Group B were classified as the adhesive failure mode. It indicated that the sandblasting treatment could slightly improve the bonding performance of the nickel surface. After the sandblasting and plasma

which demonstrated the stability of the bond line. Then tensile tests were conducted, and the results were recorded. The average bonding strength are listed in Table 5, and the variation of the bonding strength with the processing parameters is shown in Fig. 8. From Fig. 8, the processing parameters had different effects on the bonding strength, but no obvious law was observed between the strength variation and the parameters. Error margins were small for the experiments, and the small error margins reflected a high confidence level of the experimental survey. The results were then analyzed using the “Analyze Taguchi Design” function of the Minitab software. The mean response is given in Table 6, and the main effect plot is shown in Fig. 9.

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treatment, more samples in Group C showed a mixed failure mode of the adhesive failure and fibre-tear failure. However, complete debonding was still observed between the nickel plate and adhesive in some samples of Group C. With the ultrasonic vibration, it can be seen that all the samples in group D were classified as the mixed failure mode of the adhesive failure and fibre-tear failure or the mixed failure mode of the adhesive failure, fibre-tear failure and cohesive failure. The torn area of the CFRP laminate was larger, indicating that the ultrasonic action further strengthened the adhesion of the plasma-modified nickel surface, and thus the bonding strength of the CFRP/Ni joints was effectively improved.

3.3. Surface morphology analysis

In order to explore the mechanism of the bonding improvement, the surface morphology, the surface roughness and the surface wettability of the nickel plates with different surface treatments were analyzed. The nickel plates were treated according to Table 3. The surface morphology observed by the SEM is shown in Fig. 12. Fig. 12(a) shows the surface morphology of a nickel plate cleaned with acetone. The surface was relatively flat, but scratches were clearly observed, which was the brushing characteristic of the pristine nickel plate surface. Fig. 12(b) shows the surface morphology of the nickel plate treated by the sandblasting. Irregular pits were observed on the surface, which was caused by the high-speed impact of the blasted sand. The pits could effectively increase the surface roughness, enlarge the surface area, and facilitate the interfacial anchoring effect. Therefore, the bonding strength would be improved after the sandblasting. Fig. 12(c) shows the surface morphology of the nickel plate treated by the sandblasting and plasma. The etching effect of the plasma treatment was minimal, for the plasma flame temperature did not reach the melting point of the nickel (1453 °C). Thus, the plasma treatment had little effect on the surface morphology, which was similar to that in Fig. 12(b).

The AFM test was conducted, and the scanning area was 10 × 10 μm. The scanned images are shown in Fig. 13, and the measured surface roughness is listed in Table 7. $R_a$ represents the arithmetic mean
deviation of the surface roughness, $R_q$ represents the root mean square deviation, and $R_{\text{max}}$ represents the maximum height difference. Fig. 13 (a) shows the 2D and 3D morphology of the surface cleaned with acetone. The surface presented a regular groove pattern, which was consistent with the surface morphology observed in the SEM images. The surface roughnesses $R_a$ and $R_q$ were 26.4 nm and 36.0 nm respectively, and the height difference $R_{\text{max}}$ was 318 nm. Fig. 13 (b) shows the surface morphology of a nickel plate treated by the sandblasting. It can be seen that irregular pits were formed by the impact of the sand. The surface roughnesses $R_a$ and $R_q$ were 136.2 nm and 176.3 nm respectively, which was about five times higher than that in Fig. 13(a). The height difference $R_{\text{max}}$ was also significantly increased, reaching 1321 nm. The increase in surface roughness enlarged the effective contact area between the adhesive and the nickel plate. The larger the contact area is, the greater the bonding force is. In addition, the increase of the height difference $R_{\text{max}}$ could enhance the embedding effect of the pits in the adhesive to form mechanical anchoring, thereby improving the bonding strength. Fig. 13(c) shows the surface morphology of a nickel plate treated by the sandblasting and plasma modification. The surface morphology was similar to that in Fig. 13(b). The surface roughnesses $R_a$ and $R_q$ were 145.5 nm and 188.4 nm respectively, and the height difference $R_{\text{max}}$ was 1343 nm, which was close to that of Fig. 13 (b), indicating that the plasma treatment had little effect on the surface morphology and surface roughness of the nickel plates.

The contact angles of the surfaces were tested at room temperature. Because of the high viscosity (100 Pa s at 25°C), the adhesive took quite a long time (>3 h) to achieve the equilibrium state in the contact angle test. However, the pot life of the adhesive is 35 min at room temperature, so its cross-link reaction proceeded obviously during the test, which disturbed the experiment. In addition, the spreading of the adhesive on the surfaces was too slow, and the equilibrium state was not easily identified at all, which was not convenient for the experiment. Deionized water is always used as a standard test liquid, which greatly facilitates the experiment, and the test results can also show the wettability of the surfaces. Therefore, deionized water was used as the test liquid in the experiment. The results are shown in Fig. 14, and the average contact angles are listed in Table 8. The average contact angle of a nickel plate surface cleaned with acetone was 59°, and that of a nickel plate treated by the sandblasting was 60°, which was similar to the former. After the plasma modification, the average contact angle was greatly reduced to 18°. Based on the above analysis, it can be concluded

Fig. 11. Failure mode of (a) Group A, (b) Group B, (c) Group C, (d) Group D after the tensile test.

Fig. 12. SEM images of the nickel surfaces treated by: (a) cleaning, (b) sandblasting, and (c) sandblasting & plasma treating.

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that although the plasma modification had little effect on the surface morphology, it had a significant improvement on the wettability. Because the plasma treatment introduced polar groups on the surface of the nickel plate, which increased the surface free energy, the wettability was improved [21].

### 3.4. Surface composition

The nickel plates were treated according to Table 3, and the elemental composition of the surfaces obtained by the EDS test is shown in Fig. 15. Fig. 15(a) shows the elemental composition of a nickel surface cleaned with acetone. The mass fraction of Ni was 97.79 %, which was close to the value in Table 1, and the mass fraction of O was 2.21 %, which was due to slight oxidation of the nickel surface. The elemental composition of the sandblasted surface is shown in Fig. 15(b). Compared with the surface cleaned with acetone, some elements including C, Si, Ca, Fe, Al and Mg were newly observed. The main component of the abrasive is SiC, but some additives, such as Al₂O₃, Fe₂O₃, CaO and MgO, are necessary for the abrasive. The impact of the high-speed sand jet brought those elements on the surface, but the main elements were Ni, C and O. Fig. 15(c) shows the elemental composition of a nickel surface treated by the sandblasting and plasma. Compared with that treated by the sandblasting only, the content of O was greatly increased after the plasma treatment, and a new element of N appeared. These indicated that the plasma treatment introduced active groups on the surface of the nickel plate.

Powder was scraped from the nickel surfaces treated with different methods for the FTIR test. Commonly used sample preparation methods

| Surface Treatment      | $R_a$ (nm) | $R_q$ (nm) | $R_{max}$ (nm) |
|------------------------|------------|------------|---------------|
| Cleaning               | 26.4       | 36.0       | 318           |
| Sandblasting           | 136.2      | 176.3      | 1321          |
| Sandblasting & Plasma treating | 145.5     | 188.4      | 1343          |

Fig. 13. AFM images of the nickel surfaces treated by: (a) cleaning, (b) sandblasting, and (c) sandblasting & plasma treating.
in the test are the KBr pellet method and the Nujol mull method. The KBr powder is easy to absorb moisture in the air, resulting in a wide absorption peak at about 3400 cm\(^{-1}\) in the infrared spectrum, and thus the detection of the group —OH is inaccurate, as shown in Fig. 16(b). Therefore, the Nujol mull method was used to prepare samples to exclude the moisture influence. The test results are shown in Fig. 16(a).

The Nujol is a mixture of several long-chain alkanes, and its characteristic groups include —CH\(_3\) and —CH\(_2\). The peaks at 2921 cm\(^{-1}\), 2927 cm\(^{-1}\), 2852 cm\(^{-1}\) and 2854 cm\(^{-1}\) were due to the stretching vibration of C—H of the methyl and methylene, while the peaks at 1463 cm\(^{-1}\), 1464 cm\(^{-1}\) and 1377 cm\(^{-1}\) were the bending vibration absorption peaks. The peak at 721 cm\(^{-1}\) was the rocking vibration absorption peak of C—H. These were all attributed to the Nujol. By comparing the infrared spectra of the nickel surfaces treated by different methods, it can be found that there was almost no —OH on the nickel surface cleaned with acetone. After the sandblasting, a weak stretching vibration absorption peak of —OH appeared at 3556 cm\(^{-1}\). After the plasma treatment, stronger absorption peaks of —OH were observed at 3558 cm\(^{-1}\), 3614 cm\(^{-1}\) and 3649 cm\(^{-1}\), indicating that the plasma treatment introduced more —OH groups on the surface of the nickel plate.

The absorption peaks of methyl and methylene in the Nujol were very strong, which may cover the absorption peaks of other groups, interfering with the analysis of the groups. Therefore, the KBr pellet method was then used to test and analyze other groups, and the results are shown in Fig. 16(b). There was a weak stretching vibration absorption peak of Ni—O bond (460 cm\(^{-1}\)) \(^{[31–33]}\) on the nickel surface cleaned with acetone. It was due to slight oxidation of the nickel surface. After the sandblasting, the in-plane bending vibration absorption peak of O—H (1420 cm\(^{-1}\)) appeared. Combined with Fig. 16(a), it can be seen that a small number of —OH groups were introduced on the surface after the sandblasting. In addition, the stretching vibration absorption peak of C=O (1627 cm\(^{-1}\)) and the stretching vibration absorption peak of C—O (1000 cm\(^{-1}\)) were enhanced. It was because the sand contained elements such as C and O, and more C=O bonds and C—O bonds were formed owing to impact and oxidation during the sandblasting process. The stretching vibration absorption peaks of Ni—O and Ni—C at 524 cm\(^{-1}\), 468 cm\(^{-1}\) and 428 cm\(^{-1}\) were also enhanced compared with the nickel surface cleaned with acetone. After the sandblasting and plasma treatment, the in-plane bending vibration absorption peaks of O—H at 1438 cm\(^{-1}\) and 1384 cm\(^{-1}\) were enhanced, indicating that more —OH groups were introduced on the surface. Furthermore, new peaks were observed at 877 cm\(^{-1}\) and 692 cm\(^{-1}\), which were the out-of-plane bending-vibration absorption peaks of N—H. This explained why the N element was detected in the EDS test results (Fig. 15(c)). The stretching vibration absorption peak of C=O and the in-plane bending-vibration absorption peak of N—H were overlapped at 1629 cm\(^{-1}\), which were also enhanced, indicating that the active groups were introduced on the surface of the nickel plate by the plasma treatment.

From the above analysis, it can be seen that the content of —OH and —C=O on the nickel surface was slightly increased after the sandblasting. After the plasma treatment, the content of —OH and —C=O was further increased, and —NH\(_2\) was detected, indicating that active functional groups such as hydroxyl, carbonyl and amino were introduced on the surface by the plasma treatment. The active functional groups could improve the surface wettability and react with the components in the adhesive, thus enhancing the bonding strength of the joints.

However, it can be seen from Fig. 10 that the bonding improvement was slight after the plasma treatment, but the bonding strength was significantly improved after the ultrasonic vibration was applied. From the above analysis, it was considered that the plasma treatment could improve the surface wettability of the nickel plates macroscopically, facilitating spread of the adhesive on the surface. However, the viscous adhesive, which has poor fluidity, might not fit well with the irregular pits on the nickel surface at a microscopic level. The active groups introduced by the plasma treatment cannot fully contact and react with the adhesive to form enough chemical bonds. As a result, the bonding strength of the plasma-treated samples was not significantly improved. Therefore, it is necessary to further analyze the bonding interface between the nickel plate and the adhesive.

### 3.5. Interface morphology analysis

The interface marked in Fig. 7(b) was observed by the SEM, and the results are shown in Fig. 17. Fig. 17(a) shows the bonding interface of a joint without the ultrasonic process (a sample in Group C). Bubbles were seen in the adhesive layer, which would lead to a decrease in the bonding performance. The bonding interface between the adhesive and the CFRP laminate was relatively compact, while there were many holes at the bonding interface between the adhesive and the nickel plate. These holes were mainly attributed to inadequate fill of the surface pits by the adhesive owing to its poor fluidity. Consequently, the adhesive was not in good contact with the rough surface of the nickel plate. The EDS test was performed on the nickel surface with the plasma treatment, and the elemental distribution on the surface is shown in Fig. 18. It can be seen that O and N were mainly observed in the irregular pits of the surface, indicating that the active groups such as —OH, —C=O and —NH\(_2\) introduced by the plasma treatment tended to appear in these pits. It was because the plasma was more likely to accumulate in the pits. Since the adhesive could not fully fill the pits, those active groups failed to contact and bond with the adhesive, resulting in no significant improvement in the bonding strength.

Fig. 17(b) shows the bonding interface of a joint treated by the ultrasonic vibration (a sample in Group D). It can be seen that there were no bubble defects in the adhesive layer, and the bonding interface

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**Table 8**

| Surface Treatment                      | Average contact angle |
|----------------------------------------|-----------------------|
| Cleaning                               | 59\(^{\circ}\)       |
| Sandblasting                           | 60\(^{\circ}\)       |
| Sandblasting & Plasma treating         | 18\(^{\circ}\)       |

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**Fig. 14.** Contact angles of the nickel surfaces treated by: (a) cleaning, (b) sandblasting, and (c) sandblasting & plasma treating.
between the adhesive and the nickel plate was compact without obvious hole defects. Under the ultrasonic application, the adhesive fluid was subjected to strong and immediate impact from the adherend, which encouraged bubbles in the adhesive layer to burst. Furthermore, the high-frequency impact could drive the adhesive to effectively fill the micro pits on the surface and form a good anchoring effect with the nickel plate. Accordingly, the active groups in the pits of the surface could contact and react with the adhesive to form chemical bonds to improve the bonding strength effectively.

### 3.6. Interfacial chemical characterization

In order to analyze the effect of the surface treatment and ultrasonic action on interfacial chemical bonds, FTIR tests were conducted. Fig. 19 (a) shows the infrared spectra of the uncured adhesive E9309.3NA. The pink line is the infrared spectrum of the component A, which mainly

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**Table 1:** Elemental composition of the nickel surfaces treated by: (a) cleaning, (b) sandblasting, and (c) sandblasting & plasma treating.

| Element | Wt% | At% |
|---------|-----|-----|
| O       | 2.21| 7.64|
| Ni      | 97.79| 92.36|
| Total   | 100 | 100 |

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**Fig. 15.** Elemental composition of the nickel surfaces treated by: (a) cleaning, (b) sandblasting, and (c) sandblasting & plasma treating.
contains bisphenol A epoxy resin. The peak at 916 cm\(^{-1}\) is the stretching vibration absorption peak of the terminal epoxy group. The peak at 1510 cm\(^{-1}\) is the vibration absorption peak of the benzene ring in the molecule. The blue line is the infrared spectrum of the component B, which mainly contains 3,3'-[Oxybis(2,1-ethanediyloxy)]bis-1-propanamine. The peaks at 3363 cm\(^{-1}\) and 3297 cm\(^{-1}\) are the stretching vibration absorption peaks of the amino, and the peak at 1112 cm\(^{-1}\) is the absorption peak of the ether bond. The structural formulas of the two main components are shown in Fig. 19(b).

Fig. 20 shows the infrared spectra of the Ni/adhesive bonding interface. Compared with that of the uncured adhesive in Fig. 19, the epoxy group of the four groups was significantly reduced because of the cross-linking reaction of the adhesive. The reaction process is shown in Fig. 21. The epoxy group in the component A reacted with the amino group in the component B, and thus the epoxy group was consumed. However, little epoxy group was still detected at the bonding interface owing to the incomplete reaction.

In order to confirm the promoting effect of the ultrasonic vibration on the chemical reaction, the contents of the epoxy group and the ether group at the bonding interface were calculated and compared according
The content of a group can be characterized by the area of its absorption peak. However, the absolute amount and concentration of the samples cannot be accurately guaranteed to keep the same for each test, so the calculated content is not directly comparable to that of another test. Considering that the phenyl group in the adhesive did not participate in the reaction, the ratio of the absorption peak area of a specified group to that of the phenyl group was used to characterize the content of the specified group. The peak area tool of OMNIC software (V9) was used to calculate the absorption peak area of the groups, and the results are given in Table 9. In the table, Ratio-1 represents the ratio of the epoxy peak area to the phenyl’s, and Ratio-2 represents the ratio of the ether peak area to the phenyl’s. To eliminate the interference of the curing degree of the prepared adhesive, the same batch of the prepared adhesive was used for the experiments. When the adhesive was ready, the bonding experiments were finished in about 10 min. After the bonding, all the joints were kept in an oven at 82 °C for 2 h to cure the adhesive completely.

From Table 9, Ratio-1 of a sample in Group A was 10.51 %, which indicated that there was still some epoxy group left after the adhesive was cured. For a sample of Group B, Ratio-1 was slightly decreased, while Ratio-2 was slightly increased. It was because some —OH group was introduced on the nickel surface by the sandblasting, and the group could react with the epoxy group to produce the ether group. Accordingly, the content of the epoxy group decreased slightly, and that of the ether group increased slightly. For a sample of Group C, Ratio-1 decreased to 7.61 %, while Ratio-2 increased to 81.62 %. Active groups such as —OH and —NH₂ were introduced on the nickel surface by the plasma treatment, and those groups could react with the epoxy...
group in the adhesive. Thus, the content of the epoxy group was further reduced, and the content of the ether group was increased. The reaction is shown in Fig. 22. For a sample of Group D with the ultrasonic application, Ratio-1 decreased to 4.98 %, and Ratio-2 increased to 96.23 %, indicating that the epoxy group were further consumed and more ether group were formed. It was because the ultrasonic vibration promoted adequate filling of the adhesive into the micro-scale pits on the surface, and facilitated the adhesive to fully react with the active groups (—OH, —NH₂) introduced by the plasma treatment. More chemical bond was formed between the adhesive and the nickel plate, thereby further improving the bonding strength of the CFRP/Ni joints.

According to the above analysis, the effect of the ultrasonic application on the adhesive enhancement can be summarized by Fig. 23. On one hand, the high-frequency ultrasound caused strong vibration of the
adhesive, which offered a turbulent condition, thus facilitating eliminating bubbles in the layer. On the other hand, the ultrasonic application produced dynamic impingement at the interface, enhancing the contact between the adhesive and the nickel plate. The impact contact promoted the adhesive of high viscosity to effectively fill the micro-scale pits on the surface, thereby strengthening the mechanical anchoring effect. Meanwhile, the reaction probability between the functional groups in the adhesive and the active groups on the nickel surface was increased because of the high-frequency impingement at the interface. In addition, the ultrasonic vibration inevitably produced a thermal effect in the adhesive layer to promote chemical bonding at the Ni/adhesive interface. Accordingly, the bonding strength of the CFRP/Ni joints was significantly improved with the ultrasonic vibration.

4. Conclusion

The effect of ultrasonic vibration on adhesive enhancement of plasma-modified nickel surface was studied in this paper. The ultrasonic influence on the bonding strength and the failure mode of CFRP/Ni joints was first studied by experiments. Then the physical morphology and chemical composition of the adherend surface and the bonding interface were characterized to determine the strengthening mechanism. The conclusions are drawn as follows:

(1) Compared with nickel plates cleaned with acetone, the bonding strength of the CFRP/Ni joints increased by 19.6 % after the sandblasting treatment. The effect of the plasma treatment on the bonding improvement was similar to that of the sandblasting. With the plasma treatment and ultrasonic application, the bonding strength increased by 65 %.

(2) The joints without ultrasonic vibration showed the adhesive failure in the tensile test. With the ultrasonic application, all the joints showed the mixed failure. Torn area of the CFRP substrates was larger, and debonding area at the Ni/adhesive interfaces was smaller. The ultrasonic vibration further improved the bonding strength between the plasma-modified nickel plates and the CFRP laminates.

(3) Irregular pits formed by the sandblasting could effectively increase the surface roughness and the contact area between the adhesive and the nickel plate. The plasma treatment could introduce active functional groups such as hydroxyl, amino and carbonyl on the nickel surface to improve the surface wettability.

(4) The active functional groups introduced by the plasma treatment tended to appear in the irregular pits formed by the sandblasting. However, the adhesive with high viscosity and poor fluidity did not adequately fill the irregular pits on the nickel surface at a microscopic level, and thus the active groups cannot fully contact and react with the adhesive to form enough chemical bonds. Accordingly, the bonding strength with the plasma treatment was not significantly improved compared with that with the sandblasting.

(5) The ultrasonic vibration promoted the adhesive to effectively fill the micro-scale pits on the surface of the nickel plates, thereby strengthening the mechanical anchoring effect. The ultrasonic application produced dynamic impingement at the interface, enhancing the contact between the adhesive and the adherends. The reaction probability between the functional groups in the adhesive and the active groups on the nickel surface was increased, which effectively facilitated the chemical reaction at the Ni/adhesive interface to form more chemical bonds, thereby significantly improving the bonding strength of the CFRP/Ni joints.

Besides compacting the interface, the ultrasonic application promoted the chemical bonding, thereby further improving the bonding strength of the CFRP/Ni joints, which is instructive for optimization of the adhesive bonding process.

| Table 9 |
|-----------------|-----------------|-----------------|---------------|--------|--------|
|      |     |     |     |
| A    | 30.745 | 222.111 | 292.468 | 10.51 % | 75.94 % |
| B    | 39.160 | 302.003 | 392.915 | 9.97 %  | 76.86 % |
| C    | 19.587 | 209.944 | 257.229 | 7.61 %  | 81.62 % |
| D    | 10.947 | 211.552 | 219.848 | 4.98 %  | 96.23 % |

Table 9
The absorption peak area of the groups (unit:-).

Fig. 22. The reaction of the epoxy group with the active groups on the nickel surface.
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

No data was used for the research described in the article.

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