

Article

Effect of Various Nanoparticles (GaF_3 , ZnF_2 , $\text{Zn}(\text{BF}_4)_2$ and Ga_2O_3) Additions on the Activity of CsF-RbF- AlF_3 Flux and Mechanical Behavior of Al/Steel Brazed Joints

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Abstract: In this study, brazing AA6061 to Q235 steel using flame brazing was performed with 70.9 wt.% CsF-0.5 wt.% RbF-28.6 wt.% AlF_3 fluxes doped with GaF_3 , ZnF_2 , $\text{Zn}(\text{BF}_4)_2$ and Ga_2O_3 nanoparticles, matched with a Zn-15Al filler metal, and the spreadability of the filler metal and the mechanical properties of brazed joints were investigated at the same time. The results showed suitable amounts of GaF_3 , ZnF_2 , $\text{Zn}(\text{BF}_4)_2$ and Ga_2O_3 doped into the base flux could strengthen the filler metal in wetting and spreading on the surface of aluminum alloy and steel to different degrees. The suitable ranges of GaF_3 , ZnF_2 , $\text{Zn}(\text{BF}_4)_2$ and Ga_2O_3 , respectively, were 0.0075–0.01 wt.%, 0.0075–0.01 wt.%, 0.0075–0.01 wt.% and 0.009–0.01 wt.%, and the maximum spreading area was obtained via doping with GaF_3 . The shear strength of brazed joints reached the peak at 126 MPa when 0.075 wt.% GaF_3 was added. Comparative tests proved that the activity of the CsF-RbF- AlF_3 flux doped with GaF_3 was the best. The reason was that the CsF-RbF- AlF_3 - GaF_3 flux was competent in removing oxides of the base metal and decreasing the interfacial tension, in virtue of the activity of Ga^{3+} as well as F^- .

Keywords: GaF_3 ; ZnF_2 ; $\text{Zn}(\text{BF}_4)_2$; Ga_2O_3 ; flux; Zn-Al filler metal; spreadability; mechanical properties

1. Introduction

The increase in joining dissimilar metals has been prompted by the increasing demand for light weights and fuel efficiency in modern industries in recent years [1]. The emerging trend towards lightweight, high performance and emissions reduction is leading to the increasing use of multi-material hybrid structures in electric vehicles [2]. The hybrid structure of aluminum and steel are superior to conventional materials because of the high mechanical properties of steel and the excellent corrosion resistance of aluminum [3]. Naturally, the combination of aluminum alloy and steel shows huge research value and future potential, and has become the subject of many investigators [4].

However, it remains an unconquerable problem to get stable brazed joints between aluminum/metals dissimilar to steel because of the formation of brittle and hard intermetallic compounds, like Fe_xAl_y [5]. The complexity and randomness of the brazing process makes the design and development of brazing materials more complicated and time-consuming than ordinary materials [6]. Therefore, it is absolutely imperative to control the formation of Fe-Al intermetallic compounds. El-Sayed and Naka [7] found that the maximum bond shear strength of 127 MPa was obtained for an Al-steel joint brazed at 663 K with a 3 s ultrasound application time using a Zn-14Al alloy. It was reported that the shear strength of brazed joints reached the peak at 131 MPa when the Zn-15Al filler metal was added in the joining of lap joints of 6061 aluminum alloy to the 304 stainless steel via a flame brazing process, with the Zn-xAl filler metals matching the CsF-0.5 wt.%

RbF-AlF₃ flux [8]. The interfacial layer in the weld made with the Zn-15Al filler metal was comprised of (FeAl₃)Zn_x and (Fe₂Al₅)Zn_x [9].

Nanomaterials have been widely applied due to their unique properties. Joining technology at the nanometer scale has gradually developed with the popularity of nanomaterials, which have broad application prospects in the fields of electronics, aero-space, biology and health care [10]. Previous studies have indicated that [11] trace amounts of Ga₂O₃ addition in the CsF-0.5 wt.% RbF-AlF₃ flux could obviously strengthen the Zn-2Al filler metal in its wetting and spreading on the surface of 5052 aluminum alloy and Q235 low-carbon steel. One previous study discussed how the addition of GaF₃ and Ga₂O₃ nanoparticles influenced the wettability and spreadability of the CsF-AlF₃ flux under the same conditions [12].

The influence that adding heavy metal fluoride, ZnF₂, SnF₂, CdF₂, PbF₂ and KBF₄ into the KF-AlF₃ flux has on the spreadability of brazing aluminium was investigated, and adding ZnF₂ could greatly improve the brazing area [13]. Therefore, the fourth component, ZnF₂, has been considered for doping into CsF-0.5 wt.% RbF-AlF₃ flux in order to reduce the price in this paper. ZnCl₂ and SnCl₂ were added into the CsF-AlF₃ flux for connecting aluminium alloys [14]. The joints were bonded soundly when the mass fractions of ZnCl₂ and SnCl₂ are about 4%.

The effect of KBF₄ addition on the microstructure of the Mg-6Zn-1Si alloy has been investigated [15], and the morphology of the Mg₂Si phase changed with the addition of 1.5 wt.% KBF₄. To compare the activity of BF₄[−] and its unified positive ions in reducing the variables, Zn(BF₄)₂ has been chosen.

In this paper, brazing AA6061 to Q235 steel using flame brazing has been performed with improved CsF-0.5 wt.% RbF-AlF₃ fluxes doped with a GaF₃, ZnF₂, Zn(BF₄)₂ and Ga₂O₃ nanoparticles-matched Zn-15Al filler metal, and the spreadability of the filler metal and the mechanical properties of the brazed joints were investigated at the same time. XDR analysis was carried out and the reaction mechanism was analyzed. The results could be useful for brazing AA6061 to Q235 steel while choosing a suitable flux.

2. Materials and Methods

The 6061 aluminum alloy and Q235 steel were used in this work as base metals. The compositions of the base metals were listed in Tables 1 and 2. Zn-15Al alloys were chosen as the filler metal. A CsF-0.5 wt.% RbF-AlF₃ flux was prepared by using commercial CsF-AlF₃ flux and an RbF of AR purity (Zhejiang Xinrui Welding Materials Co., Ltd., Shengzhou, China). CsF-RbF-AlF₃-GaF₃ fluxes with different compositions of AR purity GaF₃ were confected and the range of GaF₃ was 0.0001–0.125 wt.%. Same amounts of ZnF₂, and Zn(BF₄)₂ of AR purity were doped in the same way to obtain CsF-RbF-AlF₃-ZnF₂ fluxes and CsF-RbF-AlF₃-Zn(BF₄)₂ fluxes. Nano Ga₂O₃ powder in the same range was doped into the CsF-RbF-AlF₃ flux to get the corresponding CsF-RbF-AlF₃-Ga₂O₃ fluxes. After mixing into a liquid, the fluxes were then dried to powder with the oven. The chemical compositions of these four nanoparticles doped into the fluxes has been listed in Table 3.

Table 1. Chemical composition of 6061 aluminum alloy (wt.%).

Alloy	Mg	Si	Cu	Cr	Mn	Zn	Al
6061	1.10	0.61	0.25	0.12	0.01	0.01	Bal.

Table 2. Chemical composition of Q235 steel (wt.%).

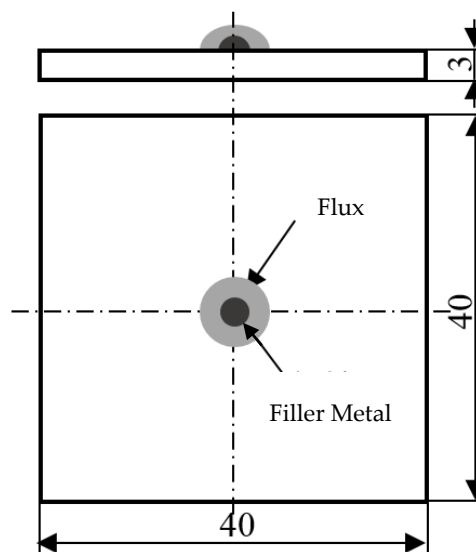
Alloy	C	Mn	Si	S	P	Fe
Q235	0.18	0.48	0.30	0.04	0.04	Bal.

Table 3. Chemical composition of nanoparticles doped into the fluxes (wt.%).

Number	1	2	3	4	5	6	7	8	9	10	11
Composition 0	0.0001	0.001	0.00325	0.0045	0.005	0.006	0.0075	0.009	0.01	0.0125	

Zn–15Al alloys were extruded into a 2 mm diameter wire as an advanced preparation. SiC paper was used for mechanically polishing the specimens and filler metals. Before brazing, these materials were all degreased with acetone and cleaned by ethanol.

In order to prepare for the spreading test, the base metals were processed into plates of 40 mm × 40 mm × 3 mm. The spreading test was performed according to China’s National Standard GB 11364-2008. The weights of the solder and flux used in the test should be 100 mg and 15 mg, respectively. The filler metals were placed in the center of the base metals covered with the fluxes prepared previously, as in Figure 1, and then put into the electrical resistance furnace (Zhejiang Xinrui Welding Materials Co., Ltd., Shengzhou, China). The heating temperature was uniformly set at 530 °C. The holding time of each test was set as 60 s. After spreading, the test boards were cleaned using ultrasonic wave waits and the spreading areas were calculated by the software Image-ro Plus (Image-Pro Plus Version 6.0). To be specific, we photographed the spreading boards with the graduated ruler and imported the image into Image-Pro Plus. By determining the actual scale in the picture and using chromatic aberration to circle the spreading outline, the spreading areas could be calculated. For the credibility of the results, the above tests of each group were repeated 5 times and the spreading areas of each group were averaged. At last, the residues of the fluxes were collected and the components in the residues were analyzed with a Brucker D8 XRD analyzer (Ningbo Institute of Materials Technology & Engineering, Ningbo, China).

**Figure 1.** Schematic diagram of the spreading test (mm).

For the shear performance tests, the supplied base metals for the brazed joint were processed into plates with the size of 60 mm × 25 mm × 3 mm. Figure 2 shows the schematic illustration of the brazed joint. The shear performance tests of the AA6061/Q235 brazed joints were carried out in strict accordance with China’s National Standard GB 11363-2008. The equipment was an SAMS-CMT5105 universal tensile testing machine (Nanjing University of Aeronautics and Astronautics, Nanjing, China), and the loading rate in the tensile process was 3 mm/min. The test results for each group of samples were all averaged from 5 samples. The brazing temperature was detected and controlled at around 530 °C.

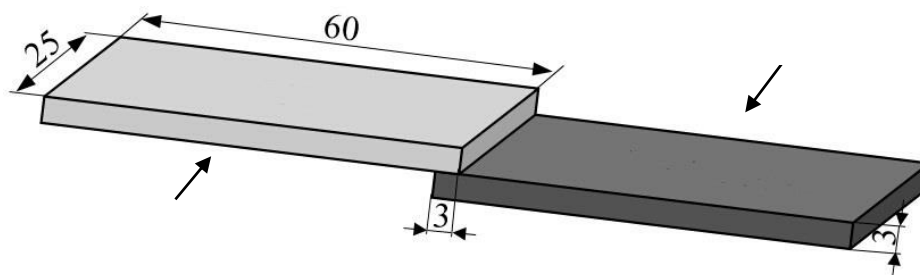


Figure 2. Schematic illustration of the brazed joint (mm).

3. Results and Discussion

3.1. The Spreadability and Wettability of Zn-15Al Filler Metal

The influence of GaF_3 , ZnF_2 , $\text{Zn}(\text{BF}_4)_2$ and Ga_2O_3 particles, doped into CsF -0.5 wt.% RbF - AlF_3 fluxes, on the spreadability of the Zn-15Al filler metal was studied via spreading tests performed on the surface of the 6061 aluminum alloy and the Q235 steel. The results were as follows.

The relationships between the concentrations of GaF_3 , ZnF_2 , $\text{Zn}(\text{BF}_4)_2$ and Ga_2O_3 and the spreading areas on the 6061 aluminum alloy are shown in Figure 3a. While the content of GaF_3 was 0.01 wt.%, the spreading area was maximized to 329 mm^2 , which was 65% larger than the area without any addition. With 0.0075 wt.%-doped ZnF_2 , the spreading area was maximized at 312 mm^2 , and showed a 56% increase compared with the results derived without adding ZnF_2 . While the content of $\text{Zn}(\text{BF}_4)_2$ was 0.0075 wt.%, the spreading area was maximized at 304 mm^2 , and was 52% larger than the area without any addition. With 0.009 wt.%-doped Ga_2O_3 , the spreading area was maximized at 321 mm^2 and showed a 61% increase compared with the results derived without adding Ga_2O_3 .

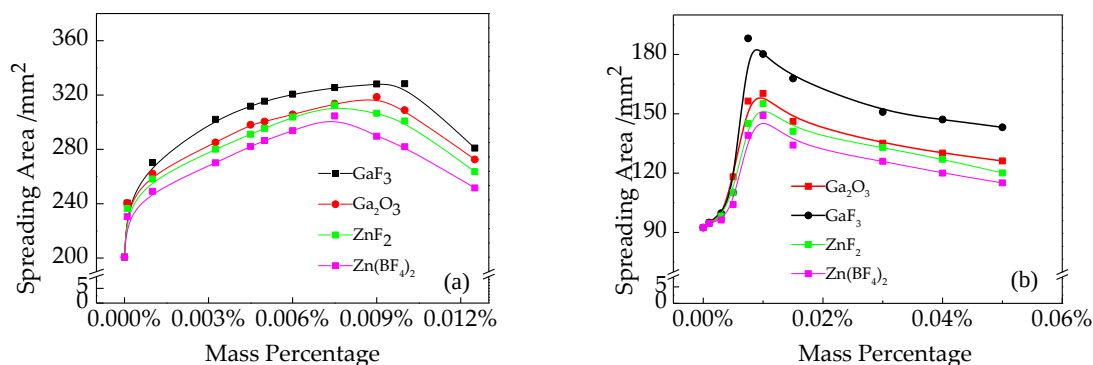


Figure 3. Spreading areas of Zn-15Al filler metal (a) 6061 aluminum alloy; (b) Q235 steel.

Figure 3b showed that the spreadability of the Zn-15Al filler metal over Q235 steel was clearly improved with GaF_3 doped into the CsF - RbF - AlF_3 flux, compared to others. While the content of GaF_3 was 0.0075 wt.%, the spreading area was maximized at 189 mm^2 . With 0.01 wt.%-doped ZnF_2 , the spreading area was maximized at 155 mm^2 . While the content of $\text{Zn}(\text{BF}_4)_2$ was 0.01 wt.%, the spreading area was maximized at 149 mm^2 . With 0.01 wt.%-doped Ga_2O_3 , the spreading area was maximized at 161 mm^2 . With the addition of GaF_3 , the spreading areas over Q235 steel underwent a 105% increase, compared with the results derived without adding GaF_3 (92 mm^2), and this substance showed clear advantages over ZnF_2 , $\text{Zn}(\text{BF}_4)_2$ and Ga_2O_3 .

The pictures of the best spreadings of Zn-15Al alloys on AA6061 and Q235, with CsF - RbF - AlF_3 flux doped with different additions, are shown in Figure 4. Taking the test results from two kinds of base metal together, it could be concluded that the suitable ranges of GaF_3 , ZnF_2 , $\text{Zn}(\text{BF}_4)_2$ and Ga_2O_3 in the CsF - RbF - AlF_3 flux respectively were 0.0075–0.01 wt.%, 0.0075–0.01 wt.%, 0.0075–0.01 wt.% and 0.009–0.01 wt.%, and the maximum spreading area of all the tests was obtained via doping with GaF_3 .

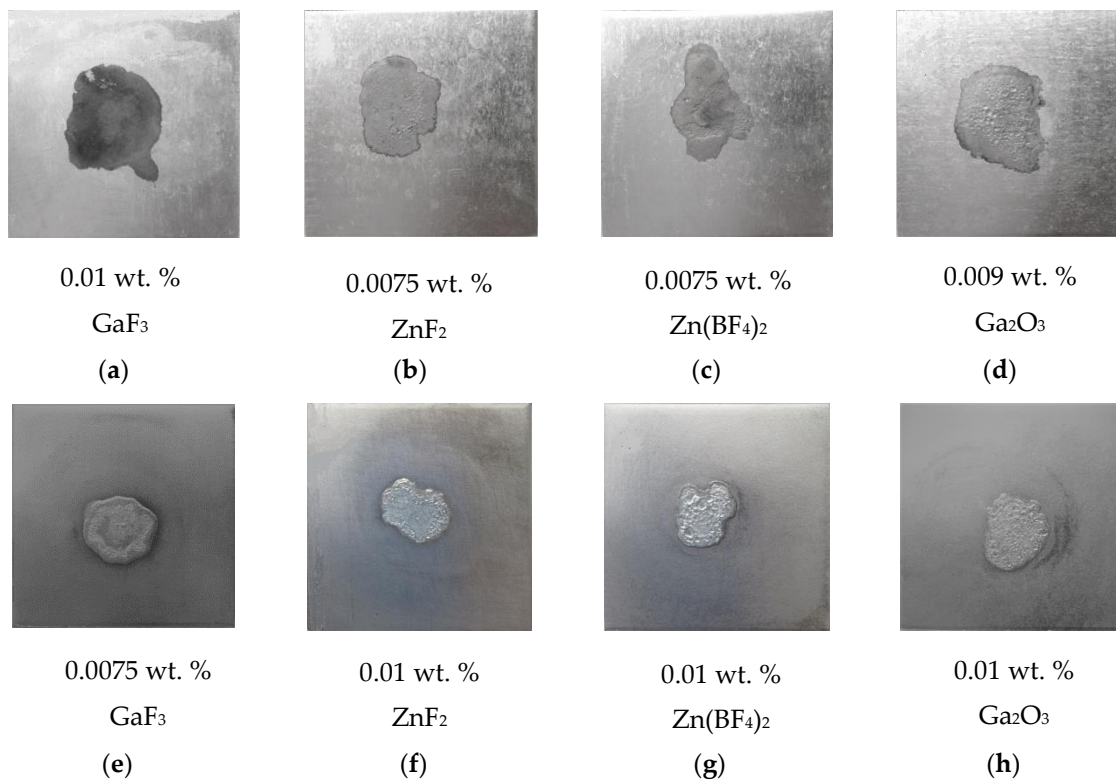


Figure 4. Spreading boards of Zn-15Al filler metal on base metals with CsF-RbF-AlF₃ flux doped with different additions: (a–d) 6061 aluminum; (e–h) Q235 low-carbon steel.

3.2. The Mechanical Properties of Brazed Joints

The variation of brazed joint shear strength was shown in Figure 5. It could be seen that the addition of GaF₃ has a significant impact on the brazed joint shear strength of AA6061/Q235. While the content of GaF₃ was 0.075 wt.%, the shear strength reached a maximum at 126 MPa, which was 110% higher than that achieved without GaF₃ addition (64.5 MPa). While the content of ZnF₂ was 0.01 wt.%, the maximum shear strength was 111 MPa. While the content of Zn(BF₄)₂ was 0.01 wt.%, the shear strength reached the top at 105 MPa. While the content of Ga₂O₃ was 0.01 wt.%, the maximum shear strength was 116 MPa.

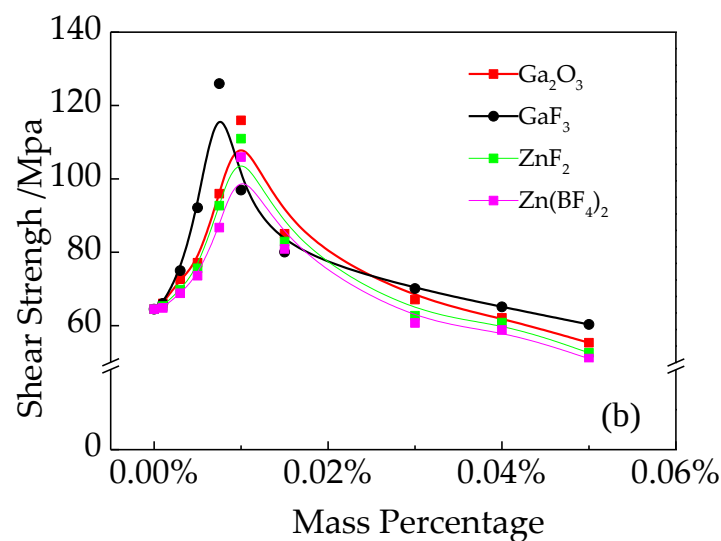


Figure 5. Influence of contents of GaF₃, ZnF₂, Zn(BF₄)₂ and Ga₂O₃ on shear strength of brazed joints.

It could be seen that, only considering the shear strength of the AA6061/Q235 brazed joints, the addition range should be controlled as 0.0075–0.01 wt.%, and this appropriate addition range coincides with the results of the spreading test. Among the four doped ingredients selected, GaF_3 showed the best performance as regards the mechanical properties of brazed joints. The second-best performance was achieved with Ga_2O_3 . At the same time, $\text{Zn}(\text{BF}_4)_2$ was inferior to others.

The typical fracture modes of the 6061/Q235 brazed joints were shown in Figure 6. The fracture of the joint occurred mainly at the interface layer of the brazing joint and the Q235 steel. It demonstrated that the interface layer between the filler metal and the Q235 steel was the weakest area of the whole joint, and that when there were layers of brittle compounds at the interface, the brazed joint would crack at the layers of brittle compounds first.

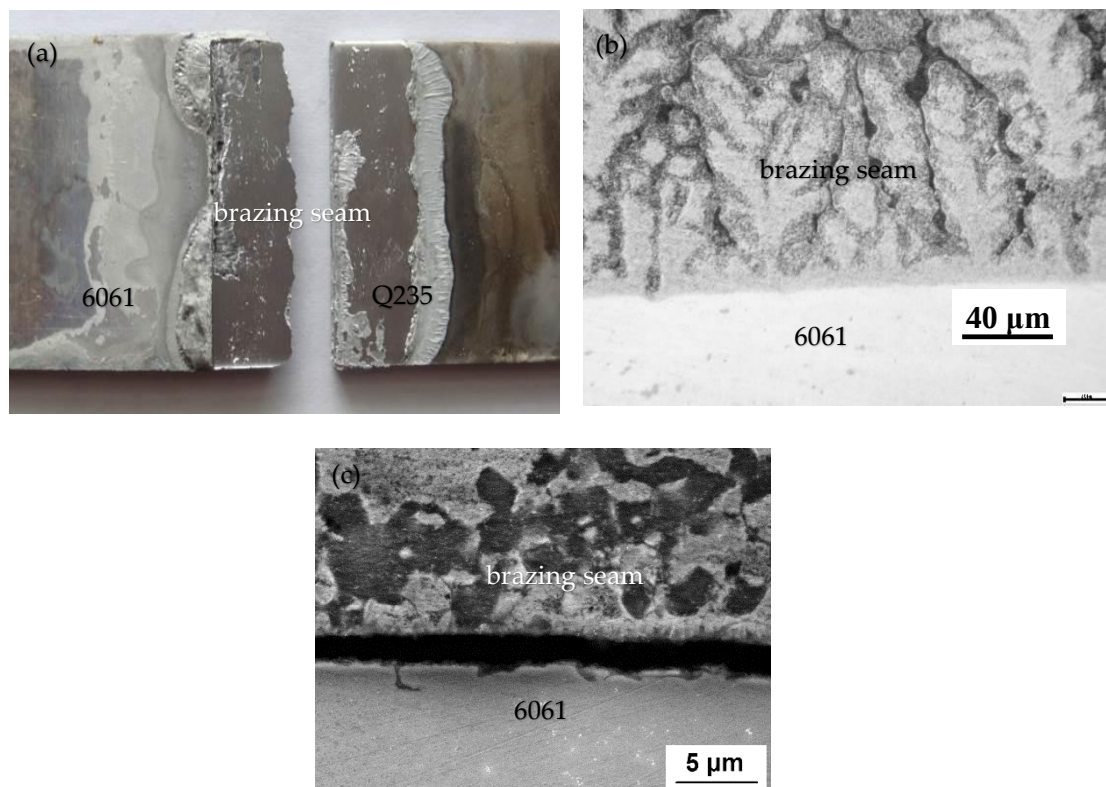


Figure 6. Typical fracture modes of 6061/Q235 brazed joints: (a) Macromorphology of fracture interface; (b) Organization of interface before shear tests; (c) Microtopography of fracture interface after shear tests.

3.3. Interfacial Effect of Different Particles

3.3.1. Effect of $\text{Zn}(\text{BF}_4)_2$

The XRD analysis results for the residues of the fluxes on Q235 low-carbon steel are shown in Figure 7. In individually evaluating the spreadability of the Zn-15Al filler metal, and the mechanical properties of the brazed joints with $\text{CsF-RbF-AlF}_3\text{-Zn}(\text{BF}_4)_2$ over the 6061 aluminum and Q235 steel, it was clear that the addition of $\text{Zn}(\text{BF}_4)_2$ significantly improved the activity of the CsF-RbF-AlF_3 flux, with a 52% increase in spreading area and a 92% increase in shear strength.



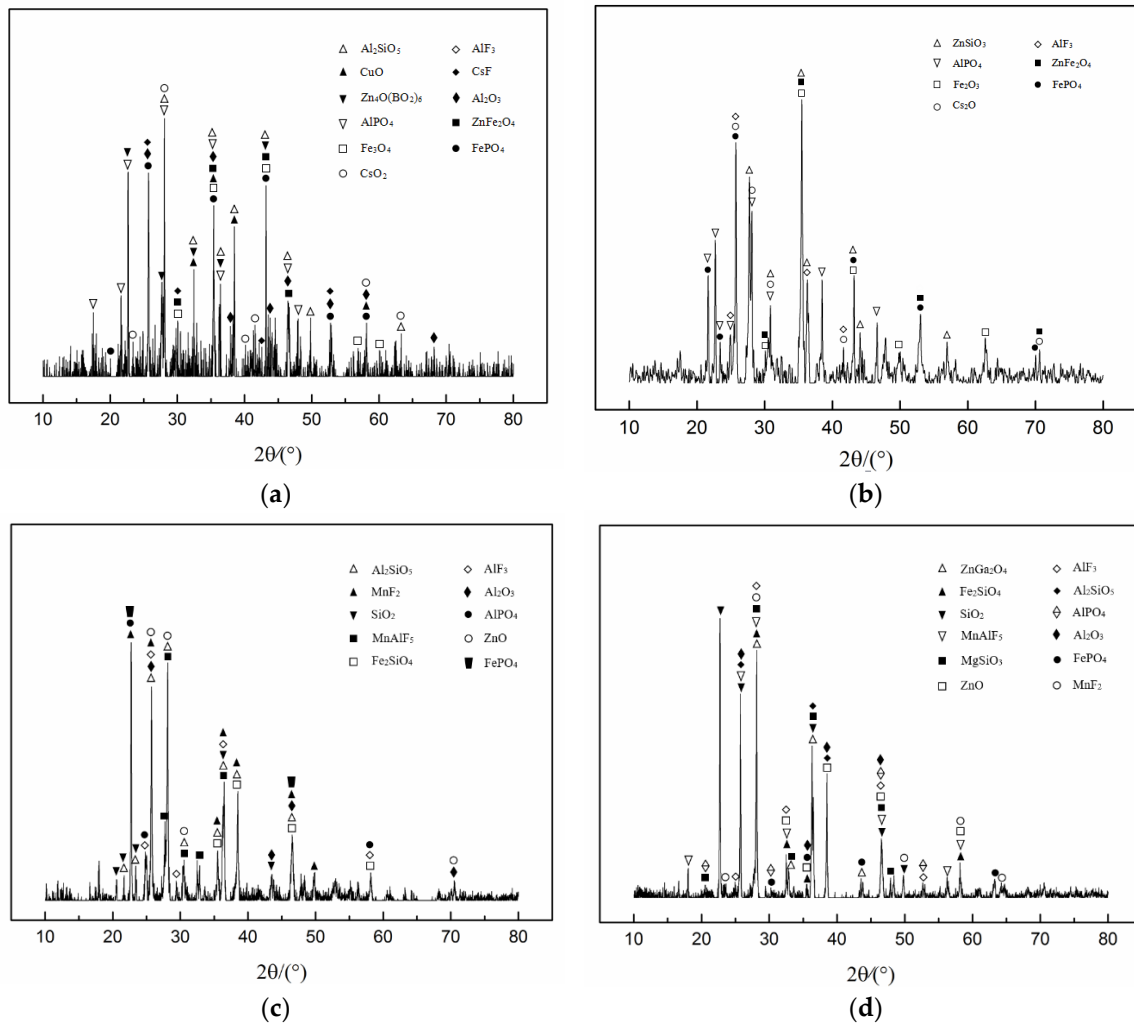


Figure 7. X-ray diffraction analysis results of the residues on Q235 low-carbon steel: (a) CsF-RbF-AlF₃-Zn(BF₄)₂; (b) CsF-RbF-AlF₃-ZnF₂ flux; (c) CsF-RbF-AlF₃-Ga₂O₃ flux; (d) CsF-RbF-AlF₃-GaF₃ flux.

Zn(BF₄)₂ could produce BF₃ as shown Equation (1). BF₃ could react with oxides such as FeO, Fe₂O, NiO, Cr₂O₃ and ZnO, thus playing the role of removing the oxide film and promoting the wetting, spreading and flowing of the molten filler metal in the brazed part [16]. However, BF₃ could not react with oxides such as Al₂O₃ and MgO on the surface of the aluminum alloy, thus limiting the activity of the CsF-RbF-AlF₃-Zn(BF₄)₂ flux.

3.3.2. Effect of ZnF₂

It was found in the study [13] that adding ZnF₂ to the flux could produce a mass transfer effect, and improve the activity of the flux. This was because the ZnF₂ was reduced by the base metal, and molten Zn has great solubility in aluminum. It was also observed that adding ZnF₂ could reduce the initial temperature of the flux, which was also related to the reduction of the precipitation of molten Zn.



When heated, ZnF_2 would react as shown Equation (2). The F^- generated by the reaction would react with the oxide on the surface of the base metals, as shown in Equations (3) and (4), respectively, thus enhancing the membrane removal effect of CsF-RbF-AlF_3 flux. The Zn atoms precipitated from the reaction would react with the aluminum atoms on the surface of the aluminum alloy and the Fe atoms on the surface of steel, and the wetting and spreading would be promoted on the surface of the base metals [17].

The presence of ZnSiO_3 , ZnFe_2O_4 , AlPO_4 and FePO_4 in the residue on the one hand indicated that the ZnF_2 in the flux did participate in the reaction, and the Zn atoms from the reduced precipitation participated in the reaction and played a role in improving the activity. On the other hand, it indicated that the enrichment phenomenon of P occurred, which resulted in the formation of compounds such as AlPO_4 and FePO_4 , which also played an important role in the removal of the oxide film from the steel surface.

3.3.3. Effect of Ga_2O_3

The “skin effect” of high-frequency currents is a well-known natural phenomenon in physics, especially in electromagnetism. However, the “skin effect” of some oxides or halides in brazing has rarely been reported. It was found that [18] adding a very small amount of Ga_2O_3 to the CsF-RbF-AlF_3 flux could increase the brazed area by about 50~90%. Observing the surface of the spreading filler metal via its spectrum showed that much Ga_2O_3 was enriched at the spreading surface and at the edge of the spreading area. Further, the relative amount of Ga_2O_3 was higher here than in the middle part, from which it could be concluded that the chemical reaction mechanism resulted from the skin effect of the Ga_2O_3 , which enabled the Ga_2O_3 to flow fast and then promote the CsF-RbF-AlF_3 flux flow, so as to improve the spreadability of the filler metal.

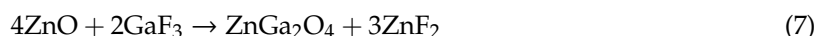


Extremely small amounts of Ga_2O_3 were enriched to participate in the reaction, and played a nonnegligible role. The appearance of MgGa_2O_4 indicated that the reaction between the flux and the MgO , which was more stable than Al_2O_3 , was also liable to react as in Equation (5). However, RbF did not appear as a compound phase in the residue, which indicated that the compound of Rb was diffused-distributed, and acted as catalysis and welding aid. CsF always appeared in the form of Cs_{11}O_3 , which indicated that CsF did undergo chemical reactions with Al_2O_3 and MgO , and replaced the O atoms in Al_2O_3 and MgO . This result explains why the spreading area increased with the increased addition of Ga_2O_3 , shown in Figure 3.

3.3.4. Effect of GaF_3

From the above it was demonstrated that the CsF-RbF-AlF_3 flux doped with GaF_3 obtained the maximum spreading area, both on 6061 aluminum alloy and Q235 steel, and reached the highest shear strength of 126MPa. The CsF-RbF-AlF_3 flux doped with GaF_3 showed remarkable superiority to that doped with ZnF_2 , $\text{Zn}(\text{BF}_4)_2$ and Ga_2O_3 .

The mechanism of GaF_3 could be summarized as a “synergistic effect” on the oxide removal of steel. When GaF_3 was doped, the flux could not only remove the oxide films on the surface of the aluminum alloy, but could also remove those on the surface of the steel at the same time, thus making it superior to brazed aluminum-steel heterogeneous materials.



The ionic compound GaF_3 consists of cations Ga^{3+} and anions F^- , and makes it such that Ga^{3+} and F^- ions can be more easily dissociated from GaF_3 than from Ga_2O_3 , as in Equation (6), while been

heated. This flux was competent in removing the oxides of the base metal and decreasing the interfacial tension, in virtue of the activity of Ga^{3+} , which has “skin effect”, as well as F^- , which reacts as in Equations (3) and (4). In addition, as Equation (7) showed, the production of ZnF_2 enhanced the activity of the flux.

4. Conclusions

It was shown that the CsF-RbF- AlF_3 flux doped with GaF_3 obtained the maximum spreading area, both on 6061 aluminum alloy and Q235 low-carbon steel, and reached the highest shear strength of 126 MPa. The above analysis led to the following conclusions:

- (1) The spreading tests indicated that the spreadability of the Zn-15Al filler metal both on Q235 steel and AA6061 alloy was promoted with a CsF-RbF- AlF_3 flux doped with GaF_3 , ZnF_2 , $\text{Zn}(\text{BF}_4)_2$ and Ga_2O_3 , and the performance of GaF_3 was the best. The suitable ranges of GaF_3 , ZnF_2 , $\text{Zn}(\text{BF}_4)_2$ and Ga_2O_3 respectively were 0.0075–0.01 wt.%, 0.0075–0.01 wt.%, 0.0075–0.01 wt.% and 0.009–0.01 wt.%.
- (2) The shear strength of the brazed joints reached its peak at 126 MPa, when 0.075 wt.% GaF_3 was added. The second-best performance was Ga_2O_3 . At the same time, $\text{Zn}(\text{BF}_4)_2$ was inferior to others.
- (3) The fluxes doped with GaF_3 were competent in removing oxides of the base metal and decreasing the interfacial tension, in virtue of the activity of Ga^{3+} , which has a “skin effect”, as well as F^- . In consequence, the activity of the CsF-RbF- AlF_3 doped with GaF_3 was better than that of others.

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