Microstructure and Corrosion Behavior of Friction Stir Welded Al Alloy Coated by In Situ Shot-Peening-Assisted Cold Spray

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Abstract

To improve the corrosion resistance of friction stir welded (FSW) joints, in situ shot-peening-assisted cold spray coating was applied to the surface of FSW joints. The microstructure and corrosion behavior of the coatings were investigated. The results showed that a dense coating was obtained by cold spraying technology. Moreover, the use of in situ shot-peening-assisted technology significantly reduced the porosity of the coating, which has a positive impact on the microstructure and the mechanical performance. Exfoliation corrosion tests showed that the coating affected positively on the corrosion resistance. On the coated surface, the craters caused by shot-peening particles were corroded in the form of exfoliation corrosion, while the exposed particles were corroded in the form of intercrystalline corrosion. This work provides a new approach to the corrosion protection of FSW joints.

Keywords Aluminum alloy coating · Cold spray · Friction stir welding · In situ shot-peening · Intergranular corrosion

1 Introduction

Aluminum alloys show excellent mechanical properties, which leads them to be used in many applications in aerospace, automobile and shipbuilding industries [1–3]. The joint quality of aluminum alloys is closely related with the welding methods. It is easy to produce weld defects by traditional fusion welding, such as porosity, thermal cracks and deformation [4]. To minimize such defects, manufacturers prefer to use solid-state joining techniques. Nowadays, friction stir welding (FSW) is widely used for welding aluminum alloys, especially 2XXX and 7XXX series [5–7]. In this technique, the heat is generated between the tool shoulder and the top of the sheets, and the heat flow is conducted from the top to the bottom of the sheets. At the same time,

there is an intense mechanical stirring through the rotation of pin tools. Although the heat input does not cause the base material to melt, a macroscopic thermo-mechanical force is generated which results in excessive microstructural inhomogeneity of the joint. Therefore, the joint is more susceptible to corrosion [8–10].

Post-weld heat treatments (PWHT) [11], micro-arc oxidation (MAO) [12] and laser surface melting (LSM) [13] have been studied to improve the corrosion resistance of FSW joints. PWHT is one of the most effective methods to improve the corrosion resistance through the immigration of the grain boundaries. At the same time, it will lead to release of tensile residual stress, which subsequently improves the mechanical properties [14, 15]. PWHT is only applied to the whole component but with limited dimensions. MAO is a proven surface modification technique used to improve the corrosion resistance of FSW joints. Yang et al. [16] showed that the corrosion resistance of the coated joints was higher than that without MAO. Similar conclusions presented by Rao et al. [17] for FSW AA2219, 2014 and 7018 joints. LSM is another surface modification method to improve the corrosion resistance of FSW joints. Kalita [18] and Padovani et al. [19] successfully implemented the LSM method on FSWs of AA2024-T351 and AA7449-T7951, respectively. It is not expected that a melting layer will form in the interface, which would cause partial delamination and elemental

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concentration. Those studies show that surface engineering is a promising direction to improve the corrosion resistance of FSW joints.

Cold spray (CS) is an emerging type of surface treatment and has developed rapidly since 1990 [20]. The CS process is a solid-state deposition process in which powder particles with a high velocity impact the samples with keeping the temperature low on the surface [21]. This process is accomplished by expanding the pressurized “hot” gas through a Laval nozzle whose temperature is below the melting point of the spray material. The temperature and deformability of the accelerated particles are increased by high-speed gas stream [22, 23]. The surfaces produce compressive residual stresses because of the bombardment of the accelerated particles. This process increases the mechanical properties of the FSW joints by relieving residual stress [24–26]. Due to these features, CS coatings are used for corrosion protection, repairing and additive manufacturing in many industries [27].

Various materials have been coated on the substrate by CS, such as aluminum alloys [28, 29], zinc alloys [30], titanium alloys [31], as well as Ni-based [32] and WC-based [33] super alloys. Continuing research suggests that CS coatings have good corrosion resistance [29, 30, 33]. The porosity is an important index to evaluate the quality of CS coating. The CS process, also referred to as the compaction effect, is based on the impact of subsequent particles on the deposited particles, which is conducive to eliminate inter-particle pores [34].

In order to further reduce the porosity, a new and innovative method has been presented by Li et al., called in situ shot-peening-assisted cold spraying [35]. In this technique, large-sized shot-peening particles (usually stainless steel) are mixed into the spray powders by intentionally introducing a tamping effect. Dense coatings of Ti6Al4V [35], Al-based [36] and Ni-based [37] powders have been successfully deposited by in situ shot-peening-assisted cold spray. This research indicated that the CS coating density can be significantly improved.

In general, CS technology is mainly applied to the surface of various materials; however, only few researchers have worked on FSW joints. Li et al. deposited an Al-based coating on FSW joint [24, 25, 38], revealing that CS is a promising method to improve the corrosion resistance of FSW joints. But the in situ shot-peening-assisted cold spray has not been applied to FSW joints. In addition, the effect of in situ shot-peening-assisted technology on the internal microstructure and coating corrosion has not been determined.

In this study, 2219 Al alloy coating was deposited on FSW AA2219-T87 by an in situ shot-peening-assisted cold spray. The microstructure and corrosion behavior of the CS coating were investigated, and the effect of in situ

shot-peening-assisted technology on the performance of CS coating was studied. Furthermore, the corrosion process of CS coating particles in exfoliation corrosion (EXCO) solution was revealed.

2 Experimental

Mixed commercial gas atomized AA2219 powders and stainless steel shot-peening (SP) powders were used as spray powders. Both particles were approximately spherical. Figure 1a shows the morphology of AA2219 powders, the average sizes of AA2219 and stainless steel particles were 5–50 μm and 150–200 μm , respectively. TEM showed that the AA2219 particles were composed of equi-axial grains ranging in size from submicron to several microns (Fig. 1b and c). The cell boundaries of the as-received AA2219 powders had strong segregation of the alloying elements. As the result of atomization, dislocations were mainly fluvial in shape. Figure 1d shows high contents of Cu, Fe and Si at the cell boundaries. In addition, the absence of oxygen in the energy dispersive X-ray spectroscopy (EDX) indicated that the powders were not oxidized. The EDS map of Cu is shown in Fig. 1e. It was clear that the Cu exists only in the form of aggregated state at the grain boundary. The stainless steel particles were mechanically mixed into the AA2219 particles to improve the impact of the coating deposition. After trying several different proportions for the composition, 60 vol% SP particles were selected. Furthermore, a spraying test without stainless steel SP particles was conducted as a comparison.

Five millimetre-thick butt-welded AA2219-T87 sheets by FSW were used as substrates for cold spraying. A welding speed of 150 mm/min and a rotational speed of 400 rpm were used during the welding. Nitrogen was adapted as an accelerating gas at temperature 350 °C under 2.8 MPa during cold spraying. The stand-off distance was 30 mm, and the traversal speed of the spray torch was set to be 60 mm/s. The powder feeder speed was 2.5 rad/min.

Both optical microscope (OM, ECLIPSE MA200, Nikon, Japan) and a scanning electron microscope SEM (SU8020, Hitachi, Japan) were used to investigate the microstructure of the surface and the cross section of the cold-sprayed coatings. To prepare the samples for these investigations, the samples were grinded, polished and etched by Dix-Keller's reagent for 15 s. The chemical composition of the coating particles was analyzed by energy dispersive spectroscopy (EDS). Transmission electron microscopy (TEM) was used to characterize the microstructure of the coatings. Vickers micro-hardness tests of CS coating and FSW were carried out to evaluate the mechanical properties, by a sclerometer (AMH-6L, ARCHIMEDES, UK). Indentations were made

Fig. 1 **a** Morphology of AA2219 powder, **b, c** TEM images, **d** EDX spectrum, **e** EDS map of a particle cross section

on the polished samples with a holding time of 10 s and a load of 200 g.

EXCO tests were applied according to the ASTM: G34-01. The cold-sprayed joint were exposed in EXCO solution (4 mol/L NaCl + 0.5 mol/L KNO₃ + 0.1 mol/L HNO₃, reagent water to make 1L at pH 0.4) for 2 h, 6 h, 24 h and 48 h, severally. The corrosion products on the samples surface were removed through the solution (50 mL H₃PO₄, 20 g CrO₃, reagent water to make 1 L) for 10 min at 90 °C according to the ASTM: G1-03. After treatment, the corrosion morphology was obtained by the digital microscope (DM, KH8700, HiRox, Japan) and SEM.

3 Results and Discussion

3.1 Microstructure

Figure 2 shows a 500-μm-thick CS coating deposited on the bottom surface of the FSW joint. Figure 2a shows different regions of the joint: weld nugget zone (WNZ), thermo-mechanically affected zone (TMAZ), heat-affected zone (HAZ) and base material (BM). The microstructure of BM was characterized by elongated grains along the rolling direction (Fig. 2b). The WNZ was composed of fine equi-axial recrystallized grains resulting from the intensive thermo-mechanical effects during the welding

process (Fig. 2d). In addition, an onion ring structure was obtained in the bottom of the WNZ, which was composed of small equi-axial grains. An elongated and crooked grain structure was presented along the rotating track in the TMAZ (Fig. 2d). In the HAZ with a thermal cycling effect (Fig. 2c), the grain structure was similar to that in the BM, but the grain size in the HAZ was larger than that in the BM.

Figure 3 shows the cross sections of the conventional CS coating and in situ shot-peening-assisted CS coating. Figure 3a shows some pores in the inner coating which was carried out without in situ shot-peening, while in Fig. 3b, which shows a denser deposited coating due to in situ shot-peening, no pores or inter-particle cracks were found in conventional CS coatings. The porosity of the conventional CS coating and in situ shot-peening-assisted CS coating was 2.56% and 0.35%, respectively. Hence, in situ shot-peening technology had a significant effect on improving the coating quality and reducing the porosity. Luo et al. [35] reported that the porosities of CS coatings declined rapidly from 14 to 0.7% with in situ shot-peening-assisted technology. A similar conclusion was proved by Wei et al. [36], who deposited Al 6061 coating by in situ shot-peening-assisted cold spraying and the porosity decreased from 14.4 to 0.4%, while the micro-hardness and corrosion resistance were obviously increased. The white dots in the FSW joints were the second phase (Al₂Cu). In the following, conventional CS coating was no

Fig. 2 Cross sections of the FSW joint after cold spraying: **a** whole view, **b** BM, **c** HAZ, **d** TMAZ/WNZ

Fig. 3 Cross sections of **a** conventional CS coating, **b** in situ shot-peening-assisted CS coating

longer studied. And all the results in this study were directed at in situ shot-peening-assisted CS coating.

Figure 4 shows the optical images of in situ shot-peening-assisted CS coating. The crescent-shaped structure was equally distributed in the coating marked by white arrows in Fig. 4a, and they were the traces left by the strikes of SP particles. A close view of the coating/substrate interface is shown in Fig. 4b, which showed no interfacial flaws such as pores and cracks. The coating particles were deeply embedded into the substrate, causing a wavy interface at a depth of 80 μm . Plastic deformation traces were induced as shown in Fig. 4c. Meanwhile, the pores were filled during particle deformation. So the coating revealed a completely dense

microstructure without inter-particle cracks except some areas on the surface coating (Fig. 4d). This result suggested that subsequent incoming particles and SP particles had a shot-peening effect on the substrate and the deposited particles. However, there was little shot-peening effect in the top area of the coating. Hence, it was easy for pores to exist in the surface layer where SP particles did not bombard.

A top view of the surface morphology of the in situ shot-peening-assisted CS coating is shown in Fig. 5a. The craters impacted by SP particles could be easily observed. The apparent diameter of the craters ranged from ~ 200 to ~ 300 μm , which was slightly larger than those of the SP particles. The particles in the craters were tamped, and an

Fig. 4 Optical images of in situ shot-peening-assisted CS coating: **a** thickness of the CS coating, **b** interface, **c** inner coating, **d** surface coating

Fig. 5 SEM images of the coating: **a** top view surface, **b** inner coating

additional hardening process was performed. When subsequent incoming particles deposited on the craters, less deformation and binging force occurred in the surface layer, so the surface particles were basically spherical. Figure 5b shows the grain microstructure of the coating. It

could be seen that the coating was divided into three kinds of characteristic grains: refined grains, elongated grains and equi-axed grains. The different grains were caused by the non-uniform plastic deformation and thermal effect. During cold spraying process, the grain boundaries of the

refined grain regions were narrowed or even discontinuous, and a large number of dislocations disappeared, which improved the corrosion resistance of the coating. Elongated grains are shown in Fig. 5b. The formation of them was associated with vertical force in the spray process [39]. Some grains were not affected by the cold spraying process and presented in the original equi-axial form.

Figure 6 shows TEM images of in situ shot-peening-assisted CS coating. Three characteristic grains are shown in Fig. 6a, which are defined in Fig. 5b. The distribution and morphology of intermetallic phases (dark region in Fig. 6) and grain boundaries were discrepant in different regions. In the region of equi-axed grains, original grains (Fig. 1c) could still be identified based on their non-deformed interior microstructure as marked by a white arrow (1). Elongated grains were marked by a white arrow (2). The grains in this region grew slightly and were elongated due to a small degree of plastic deformation and some heat input during the spraying. Grain boundaries that were longer and more continuous than those of the original grains were obtained. The segregation of the alloying elements was severe at the grain boundaries. An even more enlarged view is given in Fig. 6b, and a large degree of plastic deformation could be identified between two particles. As the result of the extrusion between particles, the size of the grains shrunk and the grain boundaries narrowed or even disappeared. Another difference was that the intermetallic phases were deformed from a fluvial shape to a capsule shape and evenly distributed in this region instead of having the segregation at the grain boundaries, as marked by white arrows (3). This was a process of refining grains, also observed by Luo et al., who analyzed the mechanism of its formation [40], which was expected to be seen during the process of cold spraying. It also could be proved from the EDS map of Cu (Fig. 6c). Compared with Fig. 1e, the distribution of Cu was significantly narrower

and more uniform, which means that the grain boundaries were narrower and the grains were refined.

Figure 7 shows a model representing the mechanism of plastic deformation during the cold spraying. Prior spraying, the powder particles were composed of equi-axial grains. While after spraying, refined grains were formed at the impact region between particles because of a large degree of plastic deformation. In the area of near refined grains, elongated grains were formed because of a small degree of plastic deformation. However, particles in some regions (white arrow 1) were not affected by the impact. The equi-axial grains could also be observed in the other areas of the coating.

3.2 Mechanical Properties

Figure 8 shows the micro-hardness results of the coated FSW joints. The hardness of the base material is 140 HV_{0.2}, while the hardness drops to 100–120 HV_{0.2} in the HAZ. The lowest value of the hardness appeared in the TMAZ, only 90 HV_{0.2}. A slight increase occurred in the WNZ, and the value was basically constant at about 110 HV_{0.2}. For the coating, the hardness values fluctuate within the range from 120 HV_{0.2} to 140 HV_{0.2}. Therefore, the surface of the coating had a micro-hardness close to the base metal. In general, the micro-hardness of the coating was obviously higher than that of the FSW joint.

The interfaces between the coating and the FSW joint presented a different view because of the changes in the micro-hardness of the FSW joint. A smooth interface was observed between the coating and the BM as shown in Fig. 9a because of the greater micro-hardness of the BM. It is difficult for AA2219 particles to strike into the BM. This phenomenon was improved in the interface between the coating and the HAZ, and a wavy interface appeared as shown in Fig. 9b. The HAZ was obviously deformed under the bombardment

Fig. 6 a, b TEM images, c EDS map of in situ shot-peening-assisted CS coating

Fig. 7 Model of plastic deformation during the spraying

Fig. 8 Micro-hardness of FSW joint

of the particles. For the TMAZ (Fig. 9c) and WNZ (Fig. 9d), the coating particles can easily strike into the substrate due to a low value of micro-hardness.

After the tensile test on the CSed FSW joint, an unexpected fracture was observed. Figure 10 shows some crescent structures equally distributed in the coating fracture.

Fig. 9 Cross sections of the interface: **a** CS/BM, **b** CS/HAZ, **c** CS/TMAZ, **d** CS/WNZ

Fig. 10 Fracture appearance of **a** in situ shot-peening-assisted CS coating, **b** inter-particle junction, **c** layered microstructure

The inter-particle binding force in the coating was mainly mechanical because cold spraying is a solid phase technology. Therefore, the fracture was formed along the junctions of the coating particles, as shown in Fig. 10b. However, a totally different fracture microstructure was obtained in the areas of the tamped SP particles. A distinct layering phenomenon caused by SP particles appeared (Fig. 10c). Numerous small uniform dimples were observed in the crescent

structures, ranging from submicron to a few microns in size (enlarged view in Fig. 10c). The bombardment of SP particles destroyed the structure of the Al particles, so the grains and grain boundaries in the Al particles were deformed. The Al particles were compressed into a whole structure and the binding force was greatly increased. Hence, the in situ shot-peening-assisted technology brought a significant improvement in the mechanical properties of the coating.

3.3 Corrosion Behavior

The cross sections of the coated FSW joint were exposed into the EXCO solution in order to compare the corrosion resistance. The morphology maps of the specimen after being immersed for 12 h are shown in Fig. 11. Blue to red areas indicated the positions from low to high, and where the position was lower, there was the severer corrosion. In the area of the BM (Fig. 11a), the corrosion morphology was mainly circular pitting pits and the maximum corrosion depth reached 103.5 μm . Negative corrosion was obtained in the HAZ/TMAZ as shown in Fig. 11b. The corrosion morphology was characterized by splats along the direction of the grain expansion. The maximum corrosion depth reached 135.5 μm . In the WNZ (Fig. 11c) and the coating (Fig. 11d), a positive corrosion situation occurred. Only a few small pitting pits were observed in those areas. The maximum corrosion depth was only up to 45.0 μm in the WNZ and 47.0 μm in the coating. The coating had a better protective effect on the FSW joint. From the coating map, the pitting pits were located in the pores of the coating. It follows that the porosity directly affected the corrosion resistance of the coating. There were no pores in the areas of the tamped SP particles because of a large degree of plastic deformation. Therefore,

the in situ shot-peening-assisted technology can effectively improve the corrosion resistance of the coating.

SEM images of the in situ shot-peening-assisted CS coating surface exposed into EXCO solution for 4 h, 12 h, 24 h and 48 h are shown in Fig. 12. Up to 24 h, the coating showed less evidence of corrosion. However, after immersing 24 h, some obvious pores occurred on the coating surface and the structure of the coating started to break down. Severe corrosion occurred on the coating surface after 48 h. The craters completely disappeared and pits were distributed on the coating surface. The pit sizes were between 100 and 500 μm . Figures 5a and 12 (craters, 4 h) show particles deposited on the craters disappeared after 4 h of corrosion. Because work hardening happened when the craters were impacted, and the craters showed a high micro-hardness, the subsequent particles had a weak bonding force onto the craters. Under the breakdown of the corrosion solutions, it is easy for the particles onto the craters to fall off.

The coating surface was composed of craters and exposed particles; therefore, the corrosion behavior of the coating was studied in two parts. Firstly, the crater began to be eroded after the coating was immersed for 12 h. Some pores appeared on the craters, and corrosion products had slight abscission marked by white arrows (1). A typical exfoliation

Fig. 11 Three-dimensional maps of corrosion morphology in different areas: **a** BM, **b** HAZ/TMAZ, **c** WNZ, **d** coating

Fig. 12 Corrosion morphologies of in situ shot-peening-assisted CS coating in EXCO solution for different immersion time

corrosion morphology was obtained after immersion for 24 h. The crater surface was characterized by layer chip, and corrosion products of the corroded crater fell off in sheets, leaving holes expanding into the inner coating, as marked by a white arrow (2). In the regions where the corrosion rate became higher, for example immersion for 48 h, corrosion media extended along the holes and the crater was diminished. The coating particles under the craters were exposed and eroded. The coating had a loose and porous microstructure; therefore, the corrosion resistance was decreased. The SEM images of the particle morphology at different corrosion times are shown in Fig. 12 (last four images). The particles began to erode during the immersion for 12 h. Corrosion occurred in the elongated grain boundaries of the particles marked by a white arrow (3). The particles experienced typical intercrystalline corrosion after immersion for 24 h. All the grain boundaries of the particle were eroded, and the particles had no structural damage. Although that had changed after 48 h of immersion, corrosion media penetrated into the inside of the particles along the grain boundaries. Numbers of particles were burst due to the growth of the corrosion products, leading to the structural damage marked by a white arrow (4). Conclusively, the coating surface was corroded in the form of exfoliation corrosion. On the other

hand, the particles were corroded in the form of intercrystalline corrosion.

4 Conclusions

1. A dense coating was obtained by cold spraying technology. The use of in situ shot-peening-assisted technology reduced the porosity of the coating, which has a positive impact on the microstructure and the mechanical performance.
2. The immersion corrosion tests in the exfoliation solution confirmed that the coating enhanced the corrosion resistance in protecting the FSW joint. Corrosion started from the craters caused by the bombardment of SP particles in the form of intercrystalline corrosion.
3. Cold spray technology is a good consideration to improve the corrosion resistance of FSW high-strength aluminum alloys.

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