Post-Print: Corrosion Properties of Multi-Pass Overlapping FSP 6061-T6 Aluminum Alloy

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Abstract

6061-T6 aluminum alloy was subjected to multi-pass overlapping friction stir processing (FSP) with air cooling and water cooling, and the corrosion performance of the nugget zone material was characterized by means of optical microscopy, scanning electron microscopy, transmission electron microscopy, immersion corrosion testing, and electrochemical corrosion testing. The results show that: after modification of the base material by FSP with these two cooling methods, the grains in the nugget zone were significantly refined and the corrosion performance was markedly improved; compared with the base material, the nugget zone exhibited higher corrosion potential, lower corrosion current density, and higher impedance; the nugget zone material processed by air-cooled FSP exhibited better corrosion performance than that processed by water-cooled FSP.

Full Text

Preamble

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Corrosion Properties of 6061-T6 Aluminum Alloy Processed by Multi-Pass Overlapping Friction Stir Processing

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Abstract

The 6061-T6 aluminum alloy was subjected to multi-pass overlapping friction stir processing (FSP) with air cooling and water cooling. The corrosion behavior of the nugget zone materials was characterized using optical microscopy, scanning electron microscopy, transmission electron microscopy, immersion corrosion testing, and electrochemical corrosion measurements. The results demonstrate that after FSP modification with both cooling methods, the nugget zone exhibited significant grain refinement and substantially improved corrosion resistance compared to the base metal. The nugget zone showed higher corrosion potential, lower corrosion current density, and greater impedance. Notably, the air-cooled FSP nugget zone demonstrated superior corrosion performance compared to the water-cooled counterpart.

Keywords: materials failure and protection, 6061-T6 aluminum alloy, friction stir processing, nugget zone, corrosion property

1. Introduction

Multi-pass overlapping friction stir processing (FSP) is a novel severe plastic deformation technique for producing ultrafine-grained materials, evolved from the friction stir welding (FSW) process developed by Professor Mishra at the University of Missouri in 1999 [1-3]. Numerous researchers [4-6] have employed multi-pass overlapping FSP to modify aluminum alloy surfaces, investigating the microstructure and mechanical properties of processed materials and confirming the technique’ s capability to produce bulk fine-grained materials. Several studies [7,8] have reported significant improvements in corrosion performance of aluminum alloys after FSP treatment. Surekha et al. [9] observed that multi-pass overlapping FSP of 2219 aluminum alloy reduced and decomposed $CuAl_2$ precipitates, thereby enhancing corrosion resistance, and inferred that this decomposition was the primary mechanism for improved corrosion performance. In this study, rolled 6061-T6 aluminum alloy plates were modified using multi-pass overlapping FSP with two cooling methods—air cooling and water cooling. The corrosion performance before and after processing was characterized through metallographic analysis, scanning electron microscopy, transmission electron microscopy, immersion corrosion testing, and electrochemical measurements to investigate the influence of microstructural evolution in the processed zone on corrosion behavior.

2. Experimental Methods

2.1 Friction Stir Processing

Multi-pass overlapping FSP was performed on 4 mm-thick 6061-T6 aluminum alloy base plates using an FSW-3LM-002 friction stir welding machine. The processing tool featured a shoulder diameter of 16 mm and a pin diameter of 4 mm with a length of 3.8 mm. The processing parameters were as follows: rotational speed of 1000 r/min, travel speed of 50 mm/min, plunge depth of 0.2 mm, and an overlap ratio of 50% (center-to-center spacing between adjacent passes of 2 mm). Both air-cooled and water-cooled processing conditions were employed.

2.2 Microstructural Characterization

Specimens from the base metal and FSP nugget zones were sectioned by wire electrical discharge machining and mounted in epoxy. After grinding and polishing, the samples were etched with Keller’ s reagent (1% HF + 1.5% HCl + 2.5% HNO_3 + 95% H_2O) and examined using a Leica DM 2500M optical microscope. For transmission electron microscopy (TEM), samples were mechanically thinned to 50 m and then twin-jet electropolished in a solution of 33% nitric acid and 67% methanol before observation with a JEM-2100 TEM.

2.3 Immersion Corrosion Testing

Intergranular corrosion and exfoliation corrosion behaviors were evaluated through immersion tests. Intergranular corrosion testing followed the GB7998-87 standard. Specimens were extracted from the FSP nugget zones, ground and polished, then immersed in a solution of 3% NaCl + 10 mL/L HCl for 24 hours at 35°C ± 2°C. After corrosion, the samples were sectioned perpendicular to the surface, polished, and examined under an optical microscope to observe intergranular corrosion morphology and measure maximum corrosion depth.

Exfoliation corrosion testing was conducted according to GB/T 22639-2008. Mounted specimens with exposed surfaces ground and polished were immersed in EXCO solution (4 mol/L NaCl + 0.5 mol/L KNO_3 + 0.1 mol/L HNO_3) for 48 hours at 25°C ± 3°C. Following corrosion exposure, specimens were immersed in concentrated HNO_3 for 30 seconds, rinsed with water, dried, and examined using scanning electron microscopy to characterize surface corrosion morphology.

2.4 Electrochemical Corrosion Testing

Potentiodynamic polarization curves and electrochemical impedance spectroscopy (EIS) were measured using a CS series electrochemical workstation in a three-electrode cell configuration with a saturated calomel reference electrode, platinum auxiliary electrode, and sample working electrode with an exposed area of 1 cm^2 in 3.5% NaCl solution. Polarization scans were performed from -1.5 V to +2 V at a scan rate of 0.01 V/s. EIS measurements were conducted over a frequency range of 10^5 to 10^{-2} Hz with a sinusoidal excitation amplitude of 10 mV.

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3. Results and Discussion

3.1 Microstructural Characterization

Figure 1a [Figure 1: see original paper] shows the optical microstructure of the T6 base metal, revealing fibrous grains aligned parallel to the rolling direction. Figures 1b and 1c present the microstructures of the air-cooled and water-cooled FSP nugget zones, respectively. At 500× magnification, grains in the air-cooled FSP nugget zone are faintly visible, whereas those in the water-cooled condition are completely unresolvable, indicating that the water-cooled FSP nugget zone possesses significantly finer grains than the air-cooled counterpart, necessitating further TEM investigation. The frictional and deformation heat generated between the tool and base metal promotes grain growth in the recrystallized nugget zone, while forced water cooling effectively suppresses this grain growth tendency.

3.2 Immersion Corrosion Behavior

Figures 2 [Figure 2: see original paper] and 3 [Figure 3: see original paper] illustrate the intergranular and exfoliation corrosion morphologies of the T6 base metal and FSP nugget zones. Figure 2a reveals severe intergranular corrosion in the T6 base metal, with extensive grain detachment from the surface layer. After 24 hours of immersion, the maximum corrosion depth reached 280 m. The corrosion propagated along grain boundaries from the surface inward, clearly revealing the base metal grain structure. Figure 3a shows extensive surface exfoliation in the T6 base metal, with deep elongated corrosion trenches formed by grain boundary dissolution.

Figure 2b demonstrates that the air-cooled FSP nugget zone exhibited substantially reduced corrosion compared to the base metal, with limited surface corrosion area and a maximum depth of only approximately 80 m. Figure 3b shows that the air-cooled FSP nugget zone displayed only minor corrosion pits, characteristic of mild pitting corrosion. The EDS spectrum of a particle about to detach from the surface (white box in Figure 3b) revealed an Al-Mg-Si composition, indicating preferential corrosion and dissolution of Mg_2Si second-phase particles in the corrosive medium.

Figure 2c shows that the water-cooled FSP nugget zone experienced more extensive surface grain exfoliation than the air-cooled condition, with a maximum corrosion depth of approximately 120 m. While its corrosion performance was inferior to the air-cooled FSP nugget zone, it remained superior to the T6 base metal. Unlike the T6 base metal, corrosion in the water-cooled FSP nugget zone initiated at surface notches and propagated inward, forming concave corrosion pits. Figure 3c presents the exfoliation corrosion morphology of the water-cooled FSP nugget zone, showing more numerous corrosion pits than the air-cooled condition, though with shallower depths than those observed in the T6 base metal.

3.3 Electrochemical Corrosion Behavior

Figure 4 [Figure 4: see original paper] shows the polarization curves for all specimens, with corresponding electrochemical parameters summarized in Table 1 . Comparison of the free corrosion potentials and corrosion current densities reveals that the T6 base metal exhibited the lowest corrosion potential and highest corrosion current density, indicating the greatest corrosion tendency and fastest corrosion rate. The air-cooled FSP nugget zone displayed a lower corrosion potential than the water-cooled condition but a smaller corrosion current density. From a thermodynamic perspective, a higher corrosion potential indicates lower corrosion tendency; thus, the air-cooled FSP nugget zone showed greater corrosion tendency than the water-cooled counterpart. However, from a kinetic standpoint, a higher corrosion current density corresponds to faster corrosion rates, indicating that the air-cooled FSP nugget zone corroded more slowly than the water-cooled condition. Overall, the electrochemical data demonstrate that the air-cooled FSP nugget zone exhibited the best corrosion performance, followed by the water-cooled FSP nugget zone, with the T6 base metal showing the poorest performance.

Figure 5 [Figure 5: see original paper] presents the electrochemical impedance spectroscopy (EIS) Nyquist plots. The EIS spectra for the T6 base metal and water-cooled FSP nugget zone consist of a high-frequency capacitive arc and a low-frequency inductive arc. The diameter of the capacitive arc reflects the surface impedance, with larger diameters indicating greater impedance and thus better corrosion resistance. The presence of an inductive arc suggests pitting corrosion induced by Cl^- attack, with larger inductive arcs indicating more severe attack. Figure 5 shows that the T6 base metal exhibited the smallest capacitive arc diameter and a pronounced inductive arc, indicating the poorest corrosion resistance. The water-cooled FSP nugget zone displayed a capacitive arc diameter intermediate between the T6 base metal and air-cooled FSP nugget zone, with a smaller inductive arc than the base metal, confirming its superior corrosion performance relative to the base metal. The air-cooled FSP nugget zone showed the largest capacitive arc without any inductive arc, demonstrating the best corrosion resistance.

3.4 Corrosion Mechanism Analysis

Corrosion performance is fundamentally determined by microstructure. Therefore, the corrosion mechanisms were investigated through detailed microstructural analysis of the three specimen conditions. Figures 6a and 6b show TEM images of the T6 base metal at different magnifications. The base metal grains were too large to capture complete grains within the TEM field of view. However, high-density dispersed precipitates and numerous dislocations were clearly visible within the grains. Grain boundaries served as preferential sites for second-phase segregation, with continuous second-phase particle distributions forming long-range, continuous anodic dissolution pathways that increased corrosion susceptibility. Corrosion could rapidly propagate along the boundaries of large grains toward the interior. Once these corrosion channels interconnected with the surface, entire grains would exfoliate, resulting in poor intergranular and exfoliation corrosion resistance in the T6 base metal [10-12].

Figures 6c and 6d present TEM images of the air-cooled FSP nugget zone at different magnifications. Dynamic recrystallization occurred in the nugget zone, producing significantly refined, equiaxed grains with high-angle boundaries approximately 2-3 m in size. The dislocation density within grains was substantially reduced, and dispersed precipitates were notably fewer than in the base metal, likely due to dissolution or coarsening of second phases during the high-temperature processing cycle. As shown in Figure 6d, only a few large spherical particles remained at grain boundaries, with second phases distributed discontinuously rather than forming continuous corrosion pathways. This microstructural modification dramatically improved intergranular and exfoliation corrosion resistance [13].

Figures 6e and 6f show TEM images of the water-cooled FSP nugget zone at different magnifications. The water-cooled FSP nugget zone exhibited even more significant grain refinement, with grain sizes of approximately 200 nm and high-angle equiaxed structures. Figure 6f reveals that while the dislocation density was substantially lower than in the base metal, the water-cooled FSP nugget zone retained the dispersed precipitate distribution characteristic of the T6 base metal, with minimal dissolution or coarsening. Fine second phases tended to segregate near grain boundaries but remained discontinuous, forming short-range, intermittent corrosion pathways. Consequently, corrosion initiation required surface notch formation followed by progressive grain-by-grain dissolution of grains in contact with the corrosive medium. In contrast, the T6 base metal’ s large grain size facilitated long, continuous corrosion channels. Therefore, while the water-cooled FSP nugget zone showed improved intergranular and exfoliation corrosion resistance compared to the T6 base metal, its performance was inferior to that of the air-cooled FSP nugget zone.

4. Conclusions

1. Rolled 6061-T6 aluminum alloy processed by air-cooled and water-cooled FSP developed refined equiaxed grain structures with different degrees of refinement. The air-cooled FSP nugget zone exhibited grain sizes of approximately 2-3 m with dissolved and coarsened second phases, whereas the water-cooled FSP nugget zone showed grain sizes of approximately 200 nm with retained, finely dispersed second-phase distribution.
2. Immersion corrosion tests and polarization curves demonstrated that the air-cooled FSP nugget zone exhibited the best corrosion performance, followed by the water-cooled FSP nugget zone, with the T6 base metal showing the poorest corrosion resistance. Multi-pass overlapping FSP technology enables effective surface modification of 6061-T6 aluminum alloy, substantially improving the corrosion performance of the base material.

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Note: Figure translations are in progress. See original paper for figures.