Properties of Al-10.78Zn-2.78Mg-2.59Cu-0.22Zr-0.047Sr Aluminum Alloy Extrusion (Postprint)

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Abstract

The microstructure and properties of Al-10.78Zn-2.78Mg-2.59Cu-0.22Zr-0.047Sr aluminum alloy extrusions under solution treatment-T652 and pre-recovery-solution treatment-T652 conditions were investigated. The results indicate that under the aging regime of 121°C$×$24 h, the pre-recovery annealing treatment effectively refines the grains (from 9.76 μm to 5.56 μm), reduces the average grain boundary angle (from 23.59° to 17.41°), significantly increases the proportion of low-angle grain boundaries (from 53% to 67%), enhances dislocation strengthening, and markedly suppresses recrystallization. Compared with the solution treatment-T652 condition, the pre-recovery-solution treatment-T652 process improves intergranular and exfoliation corrosion resistance without compromising strength (maximum intergranular corrosion depth decreased from 125.0 μm to 91.4 μm, exfoliation corrosion rating improved from EB to EA level). The tensile strength of the alloy in the pre-recovery-solution treatment-T652 state reaches 728 MPa, demonstrating that the pre-recovery annealing treatment enhances alloy strength. Dislocation strengthening and low-angle grain boundary strengthening constitute the primary strengthening mechanisms of the alloy.

Full Text

Preamble

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Properties of an Extruded Al-10.78Zn-2.78Mg-2.59Cu-0.22Zr-0.047Sr Aluminum Alloy

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Abstract

This study investigated the microstructure and properties of an extruded Al-10.78Zn-2.78Mg-2.59Cu-0.22Zr-0.047Sr aluminum alloy subjected to solid solution-T652 and pre-recovery-solid solution-T652 treatments. The results demonstrate that under aging conditions of 121°C × 24 h, pre-recovery annealing effectively refined the grain size (from 9.76 m to 5.56 m), reduced the average grain boundary angle (from 23.59° to 17.41°), significantly increased the proportion of low-angle grain boundaries (from 53% to 67%), enhanced dislocation strengthening, and substantially suppressed recrystallization. Compared with solid solution-T652 treatment, the pre-recovery-solid solution-T652 process improved intergranular and exfoliation corrosion resistance without compromising strength (maximum intergranular corrosion depth decreased from 125.0 m to 91.4 m, and exfoliation corrosion rating improved from EB to EA). The alloy’ s tensile strength reached 728 MPa in the pre-recovery-solid solution-T652 condition, confirming that pre-recovery annealing enhances alloy strength. Dislocation strengthening and low-angle grain boundary strengthening represent the primary strengthening mechanisms.

Keywords: metallic materials, high-strength aluminum alloy, pre-recovery annealing, microstructure, mechanical properties

Al-Zn-Mg-Cu series ultra-high strength aluminum alloys have become primary structural materials in aerospace and military industries due to their high specific strength and stiffness, good corrosion resistance, fatigue performance, and excellent workability [1-3]. As heat-treatable alloys, their mechanical properties are closely related to heat treatment processes. Higher Zn and Mg contents increase elemental segregation tendencies, while solution treatment enhances supersaturation of the solid solution, reduces coarse undissolved crystalline phases, strengthens aging effects, and thereby improves tensile properties [4-6]. During aluminum alloy extrusion, significant deformation energy is stored internally. During subsequent solution treatment, the high temperature and long duration provide driving force for recrystallization from this stored energy, resulting in grain coarsening that compromises comprehensive performance [5]. Introducing a pre-recovery annealing step before solution treatment can release partial deformation energy, weaken recrystallization driving forces, and thereby suppress recrystallization. This study examines the effects of pre-recovery annealing on the microstructure and properties of an extruded ultra-high strength Al-10.78Zn-2.78Mg-2.59Cu-0.22Zr-0.047Sr aluminum alloy during solid solution-T652 processing.

1. Experimental Methods

Alloy Preparation: The experimental alloy was prepared using A00 industrial pure Al (99.79%), industrial pure Zn (99.9%), industrial pure Mg (99.9%), and master alloys of Al-50%Cu, Al-4%Zr, and Al-10%Sr as raw materials. Melting was conducted in a resistance furnace at 800°C (intermediate alloy addition

→ melting → refining and degassing → slag removal → static cooling for gas evolution), followed by casting at approximately 720°C into a cast iron mold (top outer diameter: 235 mm, inner diameter: 215 mm; bottom outer diameter: 235 mm, inner diameter: 120 mm; mold height: 50 mm). The ingot weight was approximately 28 kg.

The alloy composition was analyzed using EDS (Energy Dispersive Spectrometer), with results listed in . The alloy ingot underwent multi-stage homogenization and extrusion deformation. The homogenization annealing process consisted of 400°C × 6 h + 420°C × 6 h + 440°C × 6 h + 460°C × 12 h, with an extrusion ratio of 12:1 to produce 35 mm diameter rods. Pre-recovery annealing (250°C × 24 h + 300°C × 6 h + 350°C × 6 h + 400°C × 6 h) was performed prior to solution treatment. A strengthened solution treatment (450°C × 2 h + 460°C × 2 h + 470°C × 2 h) was employed, followed by immediate water quenching and T652 treatment, with an aging regime of 121°C × 24 h.

Tensile properties were tested according to standard GB/T 228-2002 using a WDW-200G high-temperature electronic universal testing machine. Microstructural observation was conducted using a Nikon EPIPHOH 300 optical microscope. A D8 ADVANCE X-ray diffractometer was used to measure diffraction peaks and full width at half maximum (FWHM) at a scanning rate of 5°/min over a range of 30°-120° using Cu Kα radiation ($\lambda = 0.15406$ nm). Scanning electron microscopy was performed using a Zeiss Supra 55 SEM equipped with Oxford Instrument HKL EBSD system. The metallographic etchant was Graff Sargent reagent (1 mL HF + 16 mL HNO_3 + 3 g CrO_3 + 83 mL distilled water). Intergranular corrosion testing followed GB 7998-2005 [7] and ASTM G110-1992(2009) [8] standards. Exfoliation corrosion (EXCO) testing followed GB/T 22639-2008 [9] and ASTM G34-2001 [10] standards, with macroscopic morphology documented using a digital camera. Microhardness was measured using an HV-1000 tester, and electrical conductivity was tested using a 7501 eddy current conductivity meter.

2.1 Microstructure

[Figure 1: see original paper] presents the microstructures of the experimental alloy after solid solution-T652 and pre-recovery-solid solution-T652 treatments. The metallographic images reveal that pre-recovery annealing refined the grain size and produced a more uniform distribution. Graff Sargent reagent preferentially corrodes high-energy subgrain structures, rendering them dark after etching, while recrystallized grains appear white. Elemental mapping results indicate that pre-recovery annealing significantly reduced porosity and white particulate matter. EDS analysis of selected regions (results in) shows substantial non-equilibrium eutectic structures (Al-Zn-Mg-Cu) in the as-cast microstructure. The second phase at location B primarily contains Al and Cu, identified as Al_2Cu phase. These coarse second-phase particles result from high alloy content, non-equilibrium solidification during casting, and segregation of Zn, Mg, and Cu elements in the matrix. Both non-equilibrium eutectic structures and minor second-phase constituents have low melting points and can be eliminated during homogenization. The second phases at locations A and D consist mainly of Al, Cu, and Fe with low Zn and Mg content, and a Cu:Fe atomic ratio approximating 2:1, indicating Al_7Cu_2Fe inclusion phases that are insoluble excess phases. Fe atoms in the matrix reduce grain boundary migration frequency, thereby affecting recrystallization kinetics.

2.2 XRD Analysis and Dislocation Strengthening

[Figure 2: see original paper] shows the XRD spectra and FWHM values for the experimental alloy after both treatments. Comparison of Figs. 2a and 2b indicates that pre-recovery annealing has minimal effect on diffraction peak intensity ratios, suggesting little influence on crystal orientation. However, comparison of Figs. 2c and 2d reveals that the FWHM values after pre-recovery annealing exceed those without pre-recovery, indicating increased lattice strain and dislocation density.

The relationships among XRD coherent diffraction domain size (d), lattice distortion (e), FWHM (δ), Cu-Kα wavelength (λ), and diffraction peak positions ( ) can be described by functions [11]. [Figure 3: see original paper] illustrates the relationships for calculating coherent diffraction domain size and lattice strain from XRD data. The calculated results for coherent diffraction domain size (d) and lattice strain (e^2) are listed in . The data show that lattice strain in the pre-recovery annealed sample significantly exceeds that in the non-pre-recovery sample.

Dislocation density ( ) can be described by the function [12]. Using the formula, the dislocation density of the experimental alloy was calculated and summarized in , where b is the Burgers vector (b = 0.286 nm for aluminum alloys [13]). The contribution of dislocation strengthening to strength (σ) relates to dislocation density ( ) through the Taylor equation: $\sigma\ = M\alpha Gb\ ^1/^2$ [14], where M, α, and G represent the Taylor orientation factor (3.06 without texture consideration), numerical factor (0.24) [15], and shear modulus (26 GPa), respectively. The calculated dislocation strengthening contributions are listed in .

The results demonstrate that pre-recovery annealing increases both dislocation density and its strengthening contribution. This indicates that pre-recovery annealing preserves original dislocations and suppresses recrystallization. The dislocation strengthening contribution reaches approximately 72.39 MPa, representing a 22% improvement over the alloy without pre-recovery treatment.

2.3 EBSD Analysis Results and Grain Boundary Characteristics

[Figure 4: see original paper] presents EBSD microstructures, grain boundary angle distributions, and grain size distributions after both heat treatment processes. Pre-recovery treatment increased the proportion of low-angle grain boundaries and refined the grains. Compared with the non-pre-recovery sample, the grain size distribution range narrowed significantly, with reduced maximum grain size. lists the average grain size, percentages and average angles of high- and low-angle grain boundaries. Pre-recovery treatment substantially reduced average grain size and grain boundary angle (from 9.76 m to 5.56 m and from 23.59° to 17.41°, respectively), increased low-angle grain boundary fraction (from 0.53 to 0.67), and decreased their average angle (from 5.66° to 3.80°). Correspondingly, the high-angle grain boundary fraction decreased significantly, though their average angle remained essentially unchanged.

2.4 Hardness, Electrical Conductivity, and Tensile Properties

presents the electrical conductivity, hardness, tensile strength, and elongation values with and without pre-recovery treatment. Pre-recovery annealing improved electrical conductivity (26.21% IACS vs. 26.98% IACS), hardness (220.1 HV vs. 229.1 HV), and tensile strength (706 MPa vs. 728 MPa), while reducing elongation (9.8% vs. 7.0%).

2.5 Intergranular Corrosion and Exfoliation Corrosion Properties

[Figure 5: see original paper] shows the intergranular corrosion morphologies of the Al-10.78Zn-2.78Mg-2.59Cu-0.22Zr-0.047Sr alloy under different heat treatments. Intergranular corrosion is clearly evident. Measurements indicate that the maximum corrosion depth after pre-recovery annealing is 91.4 m, a reduction of 33.6 m compared with the non-pre-recovery condition, demonstrating significant improvement in intergranular corrosion resistance.

[Figure 6: see original paper] presents exfoliation corrosion morphologies after different treatments. The non-pre-recovery sample exhibits numerous pitting holes and severe surface exfoliation corrosion (EB rating). In contrast, the pre-recovery annealed sample shows minimal exfoliation in some regions and only slight layering over most of the surface (EA rating). Compared with the non-pre-recovery sample, the pre-recovery annealed specimen displays more bright gray areas—representing uncorroded aluminum alloy—indicating improved exfoliation corrosion resistance.

Two mechanisms explain intergranular corrosion: (1) structural and compositional differences between grain boundary regions and the matrix create galvanic corrosion that develops into intergranular attack; (2) dissolution of grain boundary precipitates forms an occluded corrosion environment, leading to continuous corrosion along grain boundaries. For the experimental alloy, pre-recovery treatment significantly increased low-angle grain boundaries in the solid solution-T652 condition, reducing structural and compositional differences between grain boundary regions and the matrix, thereby enhancing intergranular corrosion resistance.

Exfoliation (lamellar) corrosion evolves from intergranular corrosion, though some researchers consider it a form of stress corrosion. Exfoliation corrosion readily occurs in alloys with high intergranular corrosion sensitivity and elongated grain structures. For the experimental alloy, pre-recovery treatment improved intergranular corrosion resistance but did not substantially alter the elongated grain morphology, indicating modest improvement in exfoliation corrosion resistance.

2.6 Effect of Pre-recovery on Strengthening Mechanisms

The properties of Al-Zn-Mg-Cu aluminum alloys are influenced by numerous factors [16]:

$\sigma_{0\cdot2} = \sigma_0 + \sigma \quad + \sigma \ + \sigma \ + \sigma\mathrm{LAGB} + \sigma\mathrm{HAGB}$

where $\sigma_{0\cdot2}$ is the yield strength, σ_0 is the lattice friction stress, σ is solid solution strengthening, σ is precipitation strengthening, σ is intragranular dislocation strengthening, σLAGB is low-angle grain boundary strengthening, and σHAGB is high-angle grain boundary strengthening. Among these, σ and σLAGB have more significant effects [17, 18]:

$\sigma\mathrm{LAGB} = \mathrm{M}\alpha\mathrm{Gb}(1.5\bar{}\mathrm{LAGB/bL})^1/^2$
$\sigma\mathrm{HAGB} = \mathrm{kH\text{-}P\ L}^{-1}/^2$

where M, α, G, and b have the same meanings and values as in equations (2) and (3), ¯LAGB is the average low-angle grain boundary angle, L is the average grain size, and kH-P is the Hall-Petch coefficient (0.04 MPa $\cdot$ m^1/^{2}). The calculated strengthening contributions are listed in .

The data show that pre-recovery annealing increased the total strengthening from dislocation, low-angle grain boundary, and high-angle grain boundary mechanisms from 108.96 MPa to 132.12 MPa—a 23.16 MPa improvement. According to tensile test results, tensile strength increased by 22 MPa after pre-recovery annealing. This demonstrates that pre-recovery annealing enhances anti-recrystallization capability, preserves numerous low-angle grain boundaries and subgrain structures, suppresses recrystallization, and thereby improves alloy strength. The strength enhancement primarily originates from increased total strengthening via dislocation, low-angle grain boundary, and high-angle grain boundary mechanisms.

Conclusions

1. Pre-recovery annealing significantly refined the average grain size of the novel aluminum alloy and preserved more subgrain structures.
2. Pre-recovery annealing markedly reduced the high-angle grain boundary fraction, suppressed recrystallization during solution treatment, and increased both the proportion and strengthening effect of low-angle grain boundaries.

3. Pre-recovery annealing enhanced tensile strength by increasing dislocation density and dislocation strengthening effectiveness.
4. Pre-recovery annealing substantially improved resistance to both intergranular and exfoliation corrosion.

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