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Corrosion Behavior of Cu-7Ni-3Al-1Fe-1Mn Alloy Subjected to Different Processing Techniques

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Abstract

Copper–nickel alloys are critical materials for marine engineering, and their corrosion resistance is closely related to their microstructural states. However, the mechanism by which processing technologies regulate corrosion behavior through altering grain boundary character distribution remains unclear. In this study, the corrosion behavior of a Cu–7Ni–3Al–1Fe–1Mn alloy in 3.50 wt% NaCl solution was compared among three states: as-cast (AC), 60% deformation cross-rolled (CR), and hot-extruded (HE) with an extrusion ratio of 12.30. The aim was to reveal the key role of grain boundary character distribution in corrosion behavior. The results showed that the corrosion resistance order was HE > CR > AC, with corrosion rates of 0.0062, 0.0068, and 0.015 mm/a, respectively. CR exhibited sufficient recrystallization (83.80%) and was dominated by high-angle grain boundaries, resulting in continuous intergranular corrosion. HE showed a low degree of recrystallization (25%), but possessed high absolute densities of coincidence site lattice (CSL) grain boundaries and triple junctions; its corrosion behavior was dominated by uniform dissolution and shallow pitting, with intergranular corrosion suppressed. The key factor determining the resistance to intergranular corrosion was the absolute number (density per unit area) of CSL grain boundaries rather than their relative proportion. Processing technologies regulate grain boundary character distribution and thereby alter corrosion paths: interconnected high-angle grain boundary networks lead to intergranular corrosion, whereas dense CSL grain boundary networks lead to uniform dissolution. Hot extrusion achieved both grain refinement and optimization of the CSL grain boundary network, transforming the corrosion mode from destructive localized corrosion to uniform and safe corrosion. These findings provide theoretical support for grain boundary engineering design of copper–nickel alloys for marine engineering.

Keywords

Grain boundary character distribution; Low- Σ CSL grain boundaries; Corrosion behavior; Intergranular corrosion; Cross rolling; Hot extrusion

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Highlights

- CR: 83.8% recrystallization, high-angle grain boundaries (HAGBs)-dominated network, continuous intergranular corrosion.
- HE: 25% recrystallization, dense low- Σ CSL boundaries, suppressed intergranular corrosion.
- Absolute CSL density, not relative fraction, governs intergranular corrosion resistance.
- Dense CSL networks shift corrosion from localized attack to uniform dissolution.
- Microstructure-driven intrinsic resistance exceeds corrosion film protection.

1. Introduction

Cu-Ni alloys are widely used in marine engineering, such as heat exchangers and seawater piping systems, owing to their excellent corrosion resistance and thermal conductivity in seawater. Although they perform well in static seawater, localized corrosion — pitting, intergranular corrosion, and erosion-corrosion — still occurs periodically during long-term service, threatening the safe operation of critical equipment. Enhancing the corrosion resistance of Cu-Ni alloys while maintaining their mechanical properties is therefore a key research priority.

The corrosion resistance of Cu-Ni alloys mainly originates from a dense passive film that isolates the matrix from seawater. North *et al.*^[1] established the two-layer film theory, attributing protection primarily to the dense inner Cu_2O layer, whereas the porous outer $\text{Cu}_2(\text{OH})_3\text{Cl}$ layer offers limited protection. Subsequent studies^[2–7] showed that Ni and Fe improve film density and protectiveness by occupying cation vacancies or substituting Cu atoms in the Cu_2O lattice, thereby reducing vacancy concentration. In Al-bearing Cu-Ni-Al-Fe-Mn alloys, Al also heals passive film defects via Al_2O_3 or $\text{Al}(\text{OH})_3$ formation^[8,9]. Nevertheless, passive film formation and stability depend not only on composition but also on microstructure.

Processing is a critical means of tailoring the microstructural state of Cu-Ni alloys, affecting grain size, grain boundary character distribution, texture, and second-phase morphology. As-cast alloys exhibit coarse dendritic structures with interdendritic segregation^[10,11], and Ni_3Al and Fe-rich phases tend to segregate at grain boundaries^[12–16], forming microgalvanic corrosion channels^[17]. Cross rolling breaks down dendrites, refines grains, promotes dispersed precipitates^[18,19], and introduces residual strain^[20]. Hot extrusion disrupts coarse dendrites and segregation, producing uniform fine equiaxed grains^[21]; the formation of equiaxed grains and fiber texture is governed by dynamic recrystallization^[22–24]. At 80% strain, a dense Cu_2O film forms while porous $\text{Cu}_2(\text{OH})_3\text{Cl}$ decreases^[25]. Yang *et al.*^[26] found that in cold-rolled Cu-Ni-P alloys, 60% reduction leads to bulging nucleation, whereas above 80% shear bands become preferred nucleation sites and twin orientations are common among recrystallized grains. Hot rolling routes also affect grain boundaries and grain size. Multi-pass small reductions yield a more homogeneous grain structure^[27] and prevent low- Σ coincidence site lattice (CSL) grain boundaries from deviating under large reductions^[28]. Zhong *et al.*^[29] reported that increasing the extrusion ratio from 9:1 to 12:1 has limited effect on grain morphology but significantly increases geometrically necessary dislocation density, promoting fine-grain strengthening. For hot-extruded Cu-Ni tubes, industrial extrusion ratios typically range from 10 to 20, balancing deformation degree, extrusion load, surface quality, and die life. These systematic differences among processing routes inevitably influence the surface film structure and localized corrosion behavior.

Most existing studies focus on a single processing technique^[30,31],

and systematic comparisons of different processing routes remain scarce. In particular, for Al-containing Cu-Ni-Al alloys, whether the Al-enabled defect-healing effect via Al_2O_3 or $\text{Al}(\text{OH})_3$ ^[32] persists under different processing states remains unclear. Research systematically correlating microstructural characteristics with electrochemical corrosion behavior, passive film composition and structure, and corrosion morphology evolution is also insufficient.

Herein, a Cu-7Ni-3Al-1Fe-1Mn alloy was selected, and two processing routes were employed: cross rolling with 60% reduction and hot extrusion with an extrusion ratio of 12.30. Cross rolling effectively breaks down as-cast dendrites and promotes recrystallization while avoiding the reduction in the low- Σ CSL grain boundary fraction caused by excessive deformation. Hot extrusion falls within the commonly used extrusion ratio range for Cu-Ni alloys, ensuring sufficient deformation to refine grains and eliminate casting defects while maintaining process operability and surface quality. The corrosion behavior of as-cast (AC), cross-rolled (CR), and hot-extruded (HE) states in 3.50 wt% NaCl solution was systematically compared. Electron back scatter diffraction (EBSD) was used to analyze grain size, grain boundary character distribution (including $\Sigma 3$ twin boundary density), kernel average misorientation (KAM), and texture. Electrochemical behavior was evaluated by electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization. Corrosion damage was assessed by a 14-day immersion test combined with three-dimensional morphology observation. X-ray photoelectron spectroscopy (XPS) (Thermo Fisher Scientific, Waltham, MA, USA) was used to analyze the chemical state of the surface corrosion film. Pitting depth and density were quantitatively characterized by three-dimensional morphology, and intergranular corrosion paths were examined by scanning electron microscopy (SEM) after removal of corrosion products. This study aims to elucidate the microstructural mechanisms by which processing routes affect the corrosion resistance of Cu-Ni alloys, reveal the influence of grain boundary character distribution on corrosion behavior, and provide a theoretical basis for optimizing processing techniques for Cu-Ni alloys in marine engineering applications.

2. Experimental Materials and Methods

2.1 Material Preparation

In this study, the Cu-7Ni-3Al-1Fe-1Mn alloy was prepared by vacuum induction melting. The raw materials comprised high-purity electrolytic copper, nickel, and aluminum (99.99 wt%), together with Cu-15 wt% Fe and Cu-25 wt% Mn master alloys, and the charge was adjusted according to the measured burn-off rate of each element. After the raw materials were completely melted, the melt was held for 10 min and subsequently cast into a graphite mold of $\Phi 75 \text{ mm} \times 80 \text{ mm}$. The actual composition of the alloy was determined by inductively coupled plasma optical emission spectrometry (ICP-OES) and is listed in **Table 1**^[33]. The ingot was turned to $\Phi 70 \text{ mm}$, and the resulting as-cast specimen was designated as AC. The melting, composition determination, and ingot

processing procedures were consistent with those reported in our previous study^[33].

The cross-rolled specimen was directly cut from the ingot by wire electrical discharge machining into a plate of 12.70 mm in thickness, followed by homogenization at 950 °C for 2 h. After the holding stage, the temperature was decreased to 900 °C, and cross rolling was performed in 14 passes using a small per-pass reduction and alternating rolling directions. The reduction was distributed as follows: 0.84 mm per pass for the first five passes, 0.42 mm per pass for the intermediate seven passes, and 0.30 mm per pass for the final three passes, with soaking at 900 °C for 25 min between adjacent

passes. The final plate thickness was 5.08 mm, corresponding to a total reduction of 60%. After air cooling to room temperature, the specimen was designated as CR.

For the hot-extruded specimen, the ingot was first homogenized at 950 °C for 2 h and then extruded in a single pass to a diameter of 20 mm using an XJ-500 metal extrusion press (Wuxi Yuanchang Machinery Manufacturing Co., Ltd., Wuxi, Jiangsu, China) at an extrusion ratio of 12.3. The die was preheated to 400 °C, bitumen was used as the lubricant, and the extrusion temperature and speed were 900 °C and 5 mm/s, respectively. After air cooling to room temperature, the specimen was designated as HE.

Table 1. Actual chemical composition of the Cu-7Ni-3Al-1Fe-1Mn alloy (wt%)^[33]

	Ni	Al	Fe	Mn	Cu
Nominal Composition	7	3	1	1	Bal.
Actual Composition	7.08	3.30	1.51	1.14	Bal.

The AC, CR, and HE specimens in this study were independently prepared, and their corrosion and electrochemical data were newly measured on independent specimens rather than taken directly from our previous study^[33]. The processing correspondence between these three states and the specimens in previous studies is as follows: AC is an as-cast specimen that was not involved in our previous study^[33]; HE has the same composition and processing as the hot-extruded unannealed specimen (A1)^[33] but was separately prepared for this study; and CR was prepared based on a literature review and our previous study^[25], and this state was also not involved in our previous study^[33].

2.2 Corrosion Testing and Characterization

Static immersion tests were carried out in accordance with JB/T 7901-2023. Cylindrical specimens ($\Phi 10$ mm $\times$ 2 mm) were cut by wire electrical discharge machining from the AC specimen, perpendicular to the rolling plane of the CR specimen, and perpendicular to the extrusion direction of the HE specimen, respectively, with three parallel specimens per group. The specimens were progressively ground with W28–W7 diamond papers, rinsed with ethanol, dried, and weighed. The corrosive medium was a 3.50 wt% NaCl solution at 25 °C, and the specimens were immersed continuously for 14 d, with the solution renewed every 7 d. After immersion, corrosion products were removed according to GB/T 16545-2025: the specimens were first immersed in a 10:1 (deionized water to 37 wt% HCl) hydrochloric acid solution for 5 min, then ultrasonically cleaned, rinsed with ethanol, dried, and weighed. The corrosion rate R was calculated from Eq. (1):

$$R = \frac{8.76 \times 10^6 \times (M_0 - M_1)}{S \times T \times D} \quad (1)$$

where R represents the corrosion rate (mm/a), M_0 and M_1 denote the specimen mass before corrosion and after corrosion product removal (g), respectively, S is the exposed surface area to the corrosive medium (mm²), T is the immersion duration (h), and D is the material density (g/cm³).

The specimens immersed for 14 d were characterized as follows: the grain boundary characteristics were analyzed by EBSD, the surface morphology of the corrosion film was observed by SEM (JSM-IT100) (JEOL Ltd., Tokyo, Japan), the corrosion products were analyzed by XPS, and the surface morphology after removal of the corrosion products was examined by three-dimensional profilometry.

2.3 Electrochemical Characterization

Electrochemical tests were performed at room temperature on a CHI600D electrochemical workstation (Shanghai Chenhua Instrument Co., Ltd., Shanghai, China) using a three-electrode configuration, with three repeated measurements per specimen. Cylindrical specimens ($\Phi 11.30$ mm $\times$ 2 mm) were cut by wire electrical discharge machining from the AC specimen, perpendicular to the rolling plane of the CR specimen, and perpendicular to the extrusion direction of the HE specimen, respectively. After the oxide scale was removed with W28 abrasive paper, a copper wire was attached to the non-working surface and the specimen was cold-mounted in epoxy resin. The working surface was progressively ground with W28–W5 diamond papers, mechanically polished, rinsed with ethanol, and blow-dried. A graphite electrode served as the counter electrode, a saturated calomel electrode (SCE) as the reference electrode, and the specimen as the working electrode. Prior to testing, the specimens were pre-immersed in 3.50 wt% NaCl solution for 1 h, and the open-circuit potential (OCP) was monitored until stabilization. EIS was then measured at the stabilized OCP over a frequency range of 0.10 ~ 10⁵ Hz with an amplitude of 5 mV, and potentiodynamic polarization curves were recorded from -0.80 to 0.20 V at a scan rate of 0.01 V/s. The EIS data were fitted using ZSimpWin software Version 3.60 (AMETEK Scientific Instruments (Princeton Applied Research), Oak Ridge, TN, USA) to obtain the equivalent circuit parameters.

3. Results

3.1 Corrosion Rate Analysis

Figure 1 presents the corrosion rates of the three alloy states after continuous immersion in 3.50 wt% NaCl solution for 14 d. A lower corrosion rate indicates better corrosion resistance of the alloy. The corrosion rate of the AC specimen was 0.015 mm/a, while those of the CR and HE specimens were 0.0068 mm/a and 0.0062 mm/a, respectively. The corrosion rate of the CR specimen was slightly higher than that of the HE specimen. Since the corrosion rate varies with immersion time — generally being higher at the initial stage and reaching a stable period after the gradual formation of a protective corrosion film^[34–36] — the assessment of corrosion resistance should also take into account the morphology of the corrosion film to determine whether the alloy has reached the stable corrosion stage.

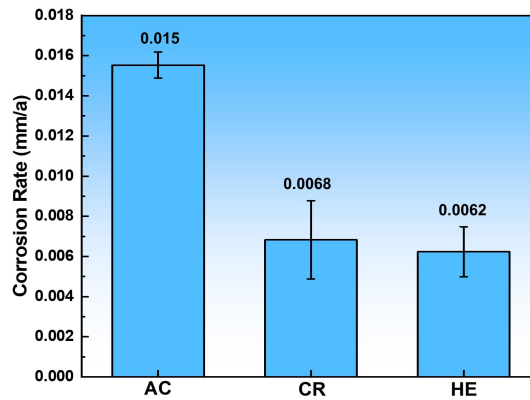


Figure 1. Corrosion rates of the three alloy states after continuous immersion in 3.50 wt% NaCl solution for 14 d. **NOTE:** AC: as-cast; CR: cross-rolled; HE: hot-extruded. Copyright: Origin 2021, PowerPoint 2016.

3.2 Microstructural Morphology of Corrosion Films

Figure 2 presents the SEM micromorphology of surface corrosion films formed on the three alloy states after 14 d of

immersion in 3.50 wt% NaCl solution. The processing states lead to distinct differences in film coverage and secondary corrosion product morphology.

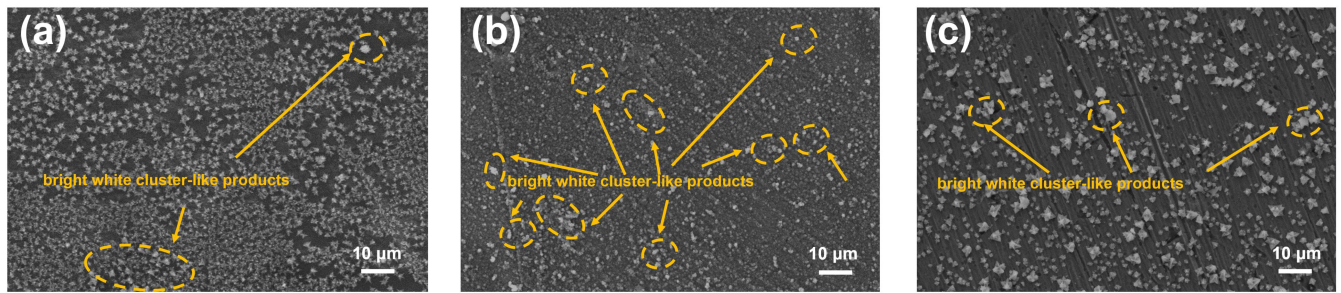


Figure 2. Micromorphology of corrosion films formed on Cu-7Ni-3Al-1Fe-1Mn alloy specimens immersed in 3.50 wt% NaCl solution for 14 d. (a) AC; (b) CR; (c) HE. Scale bar: 10 μm . Copyright: PowerPoint 2016.

Figure 2(a) shows that the corrosion film of the AC specimen mostly covers the substrate except for a few local uncovered regions, implying corrosion evolves into the later passive-film growth stage with a stabilized corrosion rate. Sparse gray polygonal star-shaped secondary corrosion products are unevenly distributed on the film surface; dense accumulation only occurs locally. No bright white clustered products are observed, and the gray products have uniform sizes without obvious coarsening or agglomeration.

For the CR specimen (**Figure 2(b)**), the substrate is fully covered by the corrosion film, indicating a stable corrosion rate. Plenty of fine gray polygonal star-shaped secondary corrosion products are uniformly spread on the film, accompanied by a small amount of bright white clustered products. Both product types are fine and homogeneously distributed, with no abnormally coarsened particles.

The HE specimen (**Figure 2(c)**) displays markedly different corrosion morphology and the lowest coverage of secondary corrosion products, leaving extensive bare substrate. After 14 d of immersion, its passive film is incomplete, and corrosion remains in the initial growth stage instead of a stable state. The surface contains gray polygonal star-shaped products and a small number of bright white clustered products. The gray products are a mixture of abundant fine dispersed particles and a few severely coarsened large particles.

3.3 Intergranular Corrosion Behavior Analysis

Figure 3 presents the intergranular corrosion morphologies of the CR and HE specimens after 14 d of full-immersion corrosion followed by removal of the surface corrosion products. A marked difference in intergranular corrosion susceptibility is observed between the two alloy states.

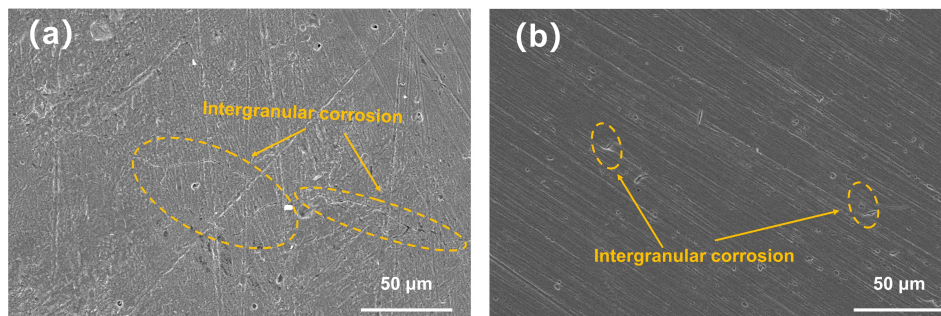


Figure 3. Surface morphologies of CR and HE specimens after descaling. (a) CR; (b) HE. Scale bar: 50 μm . Copyright: PowerPoint 2016.

The CR specimen exhibits pronounced intergranular propagation on the surface, with clear and continuous corrosion cracks along grain boundaries, indicating that this alloy state underwent relatively severe intergranular corrosion. In addition, the original mechanical polishing scratches on the surface have completely disappeared, suggesting that the CR specimen experienced sufficient uniform dissolution and that the corrosion process has entered the stable stage, which is consistent with the corrosion film morphology observed in **Figure 2(b)**. In contrast, the HE specimen surface shows only sparsely distributed short and fine corrosion cracks, without the formation of a continuous intergranular corrosion network, demonstrating its excellent resistance to intergranular corrosion. Simultaneously, fine mechanical polishing scratches are still retained on the surface, implying limited uniform dissolution and a corrosion process still in its initial stage, which is in agreement with the corrosion film morphology observed in **Figure 2(c)**.

Combined with the corrosion rate results presented in **Figure 1** (0.0068 mm/a for CR and 0.0062 mm/a for HE), it can be seen that the two alloy states exhibit similar overall corrosion rates but distinctly different corrosion modes. For the CR specimen, uniform dissolution and intergranular corrosion proceed synergistically, with intergranular corrosion contributing significantly to the total mass loss. For the HE specimen, however, uniform dissolution predominates, and intergranular corrosion is effectively suppressed.

3.4 Pitting Corrosion Behavior Analysis

Figure 4 shows the three-dimensional morphologies of the CR and HE specimens after 14 days of immersion in 3.50 wt% NaCl

solution. The deepest pits were identified along the black dashed lines in the two-dimensional plane views, with depths of $-4.37\ \mu\text{m}$ for CR and $-3.03\ \mu\text{m}$ for HE. The three-dimensional topography reveals distinct differences in pitting corrosion behavior between the two alloy states. The CR specimen exhibits fewer pits with smaller diameters but greater pit depths, whereas the HE specimen shows more pits with larger diameters but shallower depths. In addition, corrosion cracks observed in the CR specimen are irregular, short, linear features with a relatively limited number, while those in the HE specimen are more numerous and exhibit wider crack openings. This difference may be attributed to the fact that pitting behavior is closely related to the development of the corrosion film and the distribution of active sites on the substrate. As shown in **Figure 2(b)**, the corrosion film on the CR specimen surface has completely covered the substrate; the uniform and dense film effectively inhibits pit initiation in most regions, with pitting occurring only at a few weak sites. However, once pitting is initiated, since the remaining areas of the substrate are protected by the intact film and uniform dissolution is suppressed, the corrosion current is concentrated within the pit cavities, driving pit propagation in depth, resulting in small-diameter but deep pits. For the HE specimen, the corrosion film coverage is incomplete, and the inner dense Cu_2O film is susceptible to attack by the corrosive medium, rendering the substrate more prone to corrosion. Consequently, more pitting initiation sites are available; however, because uniform dissolution is not completely suppressed and the substrate dissolves concurrently over a large area, the corrosion current is dispersed, limiting the vertical propagation of individual pits and resulting in large-diameter but shallow pits.

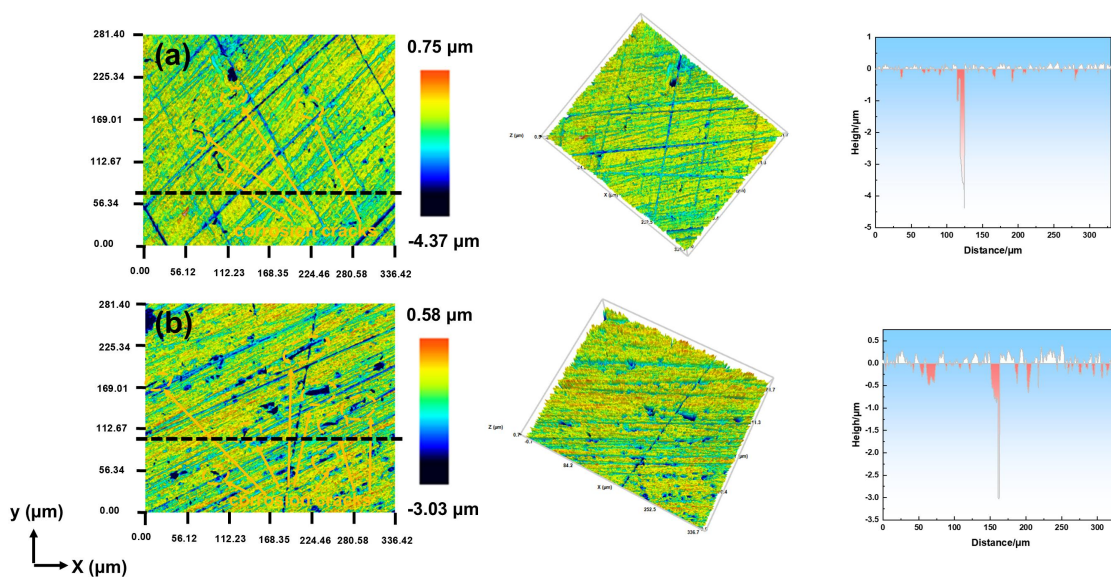


Figure 4. Three-dimensional surface topographies of CR and HE specimens. (a) CR; (b) HE. Copyright: SensoVIEW 1.9.2, PowerPoint 2016.

In combination with the intergranular corrosion morphologies shown in **Figure 3**, the corrosion behavior of the CR specimen is characterized by intergranular corrosion as the dominant mode, supplemented by deep pitting, whereas the HE specimen is dominated by uniform dissolution and shallow pitting. The hot extrusion process, through grain refinement and optimization of grain boundary character, transforms the corrosion mode from the more damaging intergranular corrosion and deep pitting to a more uniform and safer mode of uniform dissolution and shallow pitting, effectively suppressing the occurrence of localized corrosion.

3.5 Electrochemical Impedance Spectroscopy and Polarization Curves

Figure 5 shows the EIS and polarization curves of the three-state Cu-7Ni-3Al-1Fe-1Mn alloy after 14 d of immersion in 3.50 wt% NaCl solution. The EIS Nyquist plots in **Figure 5(a)** reveal that the capacitive arc radii follow the order $\text{HE} > \text{CR} > \text{AC}$, with a larger radius corresponding to superior corrosion resistance, and the hot-extruded alloy exhibiting the largest arc. Using the equivalent circuit in **Figure 5(a)**, the electrochemical interfaces of the various alloy

states were modeled with ZSimpWin software Version 3.60 (AMETEK Scientific Instruments (Princeton Applied Research), Oak Ridge, TN, USA). Given the surface inhomogeneities of the actual electrodes — including roughness and porosity — a constant phase element (CPE) was adopted in place of the ideal double-layer capacitance. In the equivalent circuit, R_s denotes the solution

resistance, R_{ct} the charge transfer resistance, and R_f the passive film resistance. The R_{ct} value reflects the barrier to charge transfer, with higher values indicating greater resistance to charge transfer and consequently lower corrosion rates. The R_f value, in turn, reflects the thickness of the surface film, with higher values corresponding to thicker films.

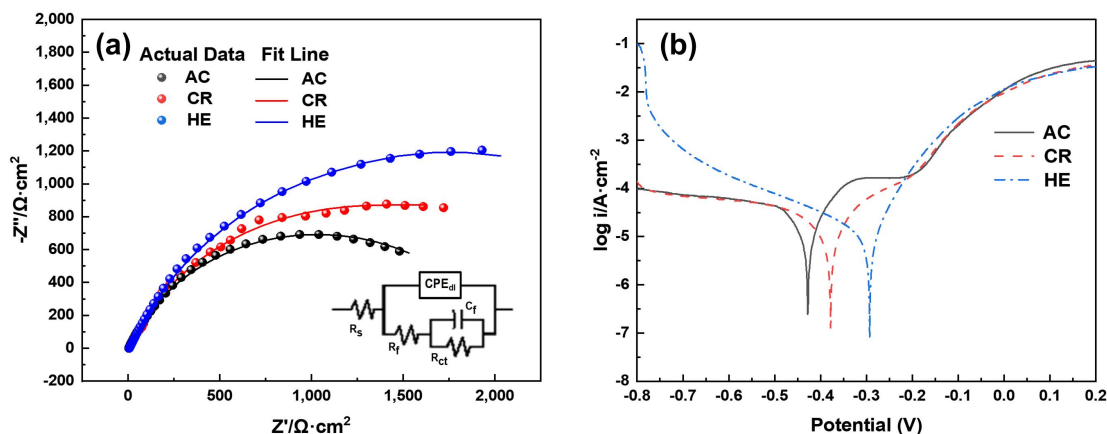


Figure 5. Nyquist and polarization curves of Cu-7Ni-3Al-1Fe-1Mn alloy after 14 d of immersion in 3.50 wt% NaCl solution. (a) Nyquist plots; (b) Polarization curves. **NOTE:** Z' : real part of impedance; Z'' : imaginary part of impedance; R_s : solution resistance; R_f : passivation film resistance; R_{ct} : charge transfer resistance; C_f : film capacitance; CPE_{dl} : constant phase element of the double layer. Copyright: Origin 2021, PowerPoint 2016.

Table 2 presents the EIS fitting results. The HE alloy exhibits an R_{ct} of 3,057 $\Omega\cdot\text{cm}^2$, exceeding those of the AC alloy (710.20 $\Omega\cdot\text{cm}^2$) and the CR alloy (1,853 $\Omega\cdot\text{cm}^2$), suggesting that hot extrusion enhances the interfacial impedance of the electrochemical corrosion

process and effectively inhibits anodic dissolution. In addition, the R_f value of the CR alloy is 2264 $\Omega\cdot\text{cm}^2$, surpassing those of the AC alloy (1,429 $\Omega\cdot\text{cm}^2$) and the HE alloy (461.90 $\Omega\cdot\text{cm}^2$), indicating that the rolling process facilitates the development of a thicker corrosion film.

Table 2. EIS fitting results

Parameter	AC	CR	HE
R_s ($\Omega\cdot\text{cm}^2$)	7.49	9.27	7.69
CPE_{dl} ($\text{F}\cdot\text{cm}^{-2}$)	2.59×10^{-4}	3.79×10^{-4}	3.22×10^{-4}
n_f ($0 < n \leq 1$)	0.74	0.74	0.75
R_f ($\Omega\cdot\text{cm}^2$)	1,429	2,264	461.90
C_f ($\text{F}\cdot\text{cm}^{-2}$)	1.32×10^{-3}	4.08×10^{-3}	8.10×10^{-3}
R_{ct} ($\Omega\cdot\text{cm}^2$)	710.20	1,853	3,057
χ^2	9.62×10^{-4}	2.81×10^{-4}	5.48×10^{-4}

NOTE: χ^2 : chi-square goodness-of-fit statistic; n_f : exponent of the CPE_{dl} .

In the polarization curves shown in **Figure 5(b)**, the Tafel extrapolation method was employed to determine the E_{corr} and i_{corr} values of the different alloys. The corrosion potential E_{corr} reflects the thermodynamic tendency of the alloy toward corrosion initiation; a more positive E_{corr} indicates a lower corrosion tendency and thus greater resistance to the onset of corrosion. The corrosion current density i_{corr} represents the corrosion current per unit area, and a lower i_{corr} value implies a weaker dissolution tendency of the alloy and consequently better corrosion resistance. As shown in **Table 3**,

the E_{corr} and i_{corr} values of the three alloy states follow the same order: the HE alloy exhibits the most positive corrosion potential and the smallest corrosion current density, followed by the CR alloy, while the AC alloy shows the most negative potential and the highest current density. This indicates that the hot extrusion process reduces the corrosion dissolution rate of the alloy in 3.50 wt% NaCl solution. This kinetic improvement is consistent with the trend of charge transfer resistance R_{ct} obtained from EIS fitting (HE > CR > AC).

Table 3. E_{corr} and i_{corr} values of the alloys

	E_{corr} (V/SCE)	i_{corr} (A/cm ²)
AC	-0.428	3.99×10^{-5}
CR	-0.379	2.18×10^{-5}
HE	-0.296	1.24×10^{-5}

4. Discussion

4.1 Microstructural Characteristics of the Alloys under Different Processing States

The microstructural differences between the two processed

states arise from their respective thermodynamic and kinetic conditions. The as-cast microstructure contains coarse dendrites and interdendritic segregation, which are structural vulnerabilities for subsequent corrosion^[37,38]. **Figure 6** compares the EBSD results of the CR and HE specimens.

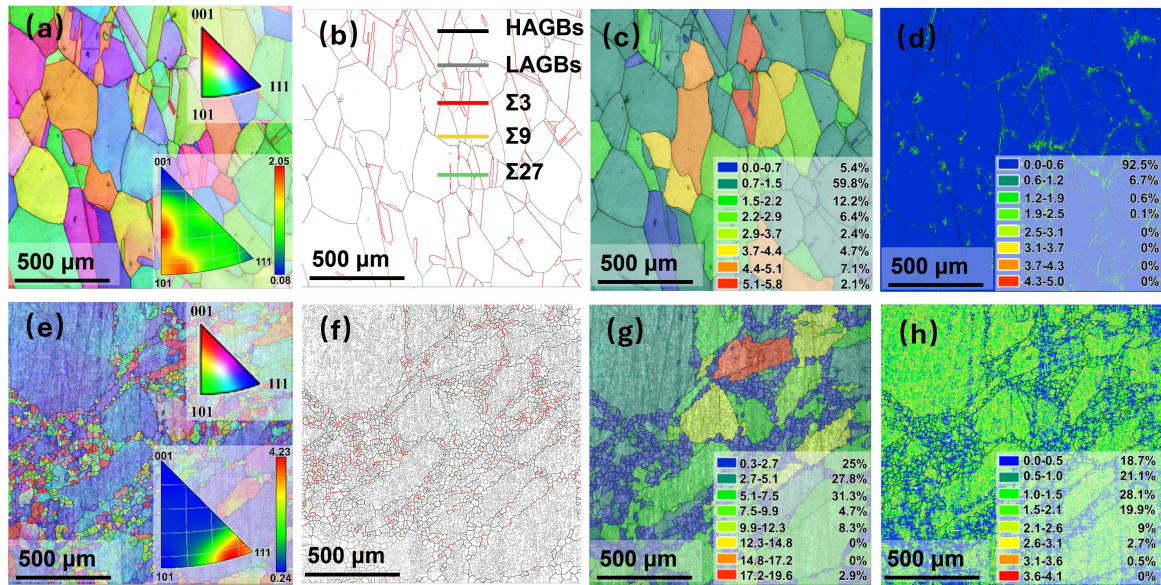


Figure 6. Microstructural characteristics of CR and HE alloys. (a) Inverse pole figure (IPF) map of CR; (b) Random boundary network map of CR; (c) Grain orientation spread (GOS) map of CR; (d) Kernel average misorientation (KAM) map of CR; (e) IPF map of HE; (f) random boundary network map of HE; (g) GOS map of HE; (h) KAM map of HE. **NOTE:** HAGBs: high-angle grain boundaries; LAGBs: low-angle grain boundaries. Scale bar: 500 μm . Copyright: AZtecCrystal 2.1, PowerPoint 2016.

In the inverse pole figure (IPF) maps (**Figure 6(a)**, **Figure 6(e)**), CR exhibits a predominant $\langle 101 \rangle$ orientation, equiaxed grains, and a uniform size distribution without obvious bimodal characteristics. HE shows a dominant $\langle 111 \rangle$ orientation and a pronounced bimodal grain size distribution, with coarse grains surrounded by fine recrystallized grains. This bimodal structure is attributed to incomplete dynamic recrystallization during hot extrusion: the severe strain from the large extrusion ratio causes local regions to reach the critical stored energy for recrystallization and nucleate and grow, while other regions with insufficient stored energy remain as deformed structures or undergo limited recovery.

In the random grain boundary network maps (**Figure 6(b)**, **Figure 6(f)**), CR is dominated by high-angle grain boundaries (HAGBs) with an extremely low fraction of low-angle grain boundaries (LAGBs). Low- Σ CSL boundaries are sparse, and CSL-related triple junctions are scarce. During cross rolling, the multi-pass, small-reduction process induces sufficient recrystallization, and grain boundary migration and reconstruction form a stable HAGBs-dominated network. HE consists primarily of LAGBs, which are densely distributed within coarse grains and at grain boundary intersections. Low- Σ CSL boundaries in HE are mainly concentrated at fine-grain positions, where they are densely distributed and interconnected, forming numerous triple junctions.

In the GOS maps (**Figure 6(c)**, **Figure 6(g)**), CR exhibits 83.80% recrystallized, 14.10% recovered, and only 2.10% deformed grains, indicating that cross rolling fully activates recrystallization and almost eliminates the deformed structure. HE shows only 25% recrystallized, 27.80% recovered, and 47.20% deformed grains, implying that nearly half of the grains retain pronounced deformed substructures and the recrystallization degree is much lower than that of CR. This is consistent with the bimodal distribution in the IPF maps and the

predominance of LAGBs, confirming the microstructural heterogeneity and incomplete recrystallization of HE.

In the KAM maps (**Figure 6(d)**, **Figure 6(h)**), pronounced misorientation is detected only near LAGBs for both specimens, while no significant misorientation is observed within grain interiors or on both sides of HAGBs, indicating that dislocations are mainly concentrated at LAGBs. Due to the large number of LAGBs in HE, the proportion of regions with high misorientation in the KAM map is significantly higher than that in CR, suggesting a higher density of retained dislocation structures in HE.

Figure 7 presents the fractions of low-angle grain boundaries (LAGBs), average grain sizes, and the fractions of low- Σ CSL boundaries and twin boundaries for the CR and HE specimens. The LAGBs fraction of the CR specimen is 9.35% (**Figure 7(a)**), and its average grain size is 284.83 μm (**Figure 7(b)**). The LAGBs fraction of the HE specimen is 76.57% (**Figure 7(c)**), and its average grain size is 210.04 μm (**Figure 7(d)**). In **Figure 7(e)**, although the CR specimen exhibits a relatively higher proportion of low- Σ CSL grain boundaries, the random grain boundary network maps in **Figure 6** clearly show that the actual number of low- Σ CSL grain boundaries in the CR specimen is still lower than that in the HE specimen. This is because the CR specimen, having undergone sufficient recrystallization, exhibits coarse and uniform grains with a relatively low total grain boundary length per unit area; thus, even with a higher CSL fraction, the absolute population remains limited. In contrast, the HE specimen contains a large number of fine grains and subgrain boundaries, resulting in an extremely high total grain boundary density; therefore, although the CSL fraction is relatively low, the absolute population of low- Σ CSL grain boundaries is still considerably higher than that of the CR specimen.

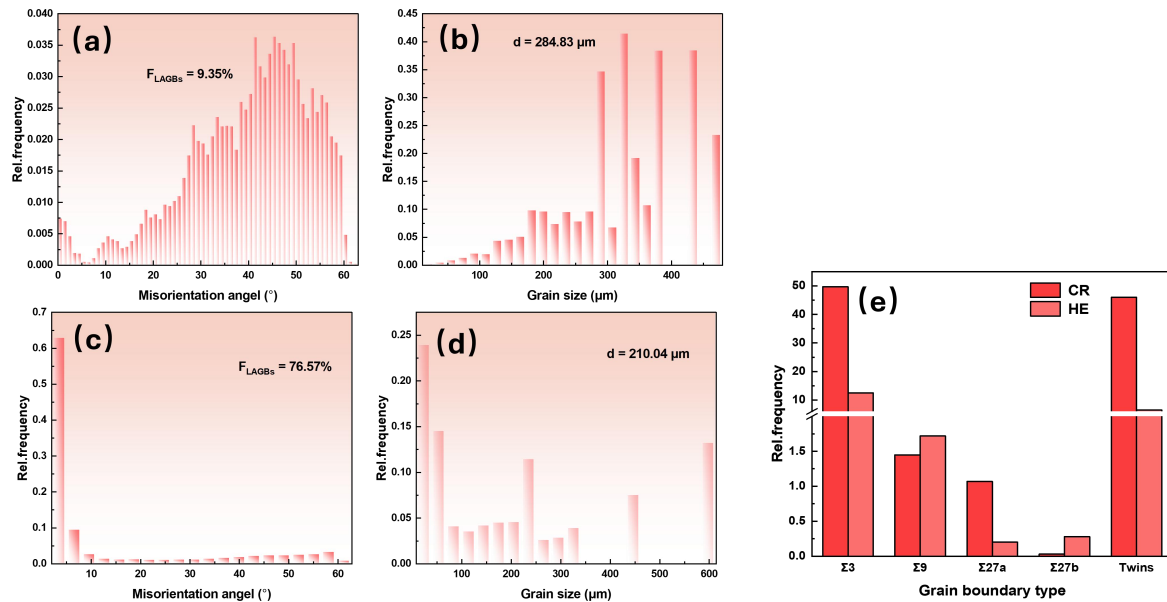


Figure 7. Grain boundary characteristics and grain size distribution of the CR and HE specimens. (a) The fraction of low-angle grain boundaries (LAGBs) in the CR alloy; (b) The average grain size of the CR alloy; (c) The fraction of low-angle grain boundaries (LAGBs) in the HE alloy; (d) The average grain size of the HE alloy; (e) Proportions of low- Σ CSL grain boundaries and twin boundaries in CR and HE alloys. **NOTE:** $\Sigma 3$: Coincidence site lattice (CSL) $\Sigma 3$ grain boundary; $\Sigma 9$: Coincidence site lattice (CSL) $\Sigma 9$ grain boundary; $\Sigma 27a$: Coincidence site lattice (CSL) $\Sigma 27a$ grain boundary, corresponding to a 31.6° rotation about the $\langle 110 \rangle$ axis; $\Sigma 27b$: Coincidence site lattice (CSL) $\Sigma 27b$ grain boundary, corresponding to a 35.4° rotation about the $\langle 210 \rangle$ axis. Copyright: Origin 2021, PowerPoint 2016.

4.2 Role of Grain Boundary Character in Regulating Corrosion Behavior

The initiation and propagation of intergranular corrosion depend on the connectivity of corrosion paths along grain boundaries. **Figure 8** schematically illustrates how characteristic grain boundaries hinder corrosion propagation. Ordinary HAGBs, with high boundary energy

and large atomic misfit, are preferentially dissolved in Cl^- -containing media and serve as primary corrosion paths. In contrast, low- Σ CSL boundaries, with low energy and good atomic coherency, are much more resistant to Cl^- attack. As shown in **Figure 8**, corrosion propagates along HAGBs but is impeded upon encountering low- Σ CSL boundaries, thereby interrupting propagation.

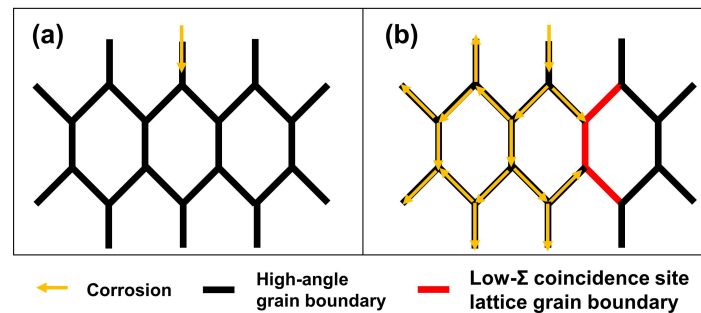


Figure 8. Schematic diagram illustrating the mechanism of characteristic grain boundaries hindering corrosion propagation. (a) Corrosion initiation; (b) Corrosion propagation. Copyright: PowerPoint 2016.

In CR, the HAGBs-dominated random grain boundary network is highly connected, forming through-channels, while the sparsely distributed low- Σ CSL boundaries are too far apart to disrupt the continuity of ordinary boundaries. Once intergranular corrosion initiates, it propagates continuously through the HAGBs network, forming through-going intergranular cracks (**Figure 3(a)**). This is the structural origin of the severe intergranular corrosion in CR.

HE exhibits the opposite behavior. Although its relative fraction of low- Σ CSL boundaries is lower than that of CR, the absolute population is higher and more densely distributed, with numerous interconnected CSL-related triple junctions. These boundaries are concentrated in fine recrystallized grain regions, effectively disrupting the connectivity of the random HAGBs network and preventing through-going corrosion channels. Even if corrosion initiates at localized grain boundaries, its propagation is

blocked by the dense low- Σ CSL boundaries ahead. This is the structural mechanism by which intergranular corrosion is significantly suppressed in HE (**Figure 3(b)**).

Pitting initiation depends on active sites on the surface film, such as inclusions, second-phase particles, and grain boundaries. In CR, the fully recrystallized structure has homogeneous, coarse grains, a low grain boundary length per unit area, and few active sites, resulting in a low probability of pit initiation. However, once pitting initiates, corrosion has entered the stable stage with complete film coverage, so uniform dissolution is suppressed and the corrosion current concentrates in the limited pits, driving them deeper. This produces the few-but-deep pitting morphology in **Figure 4(a)**.

In HE, the large fraction of deformed structures (47.20%) and high density of LAGBs provide substantially more metastable active sites than in CR, favoring extensive pit initiation and a

larger number of pits. Nevertheless, because corrosion in HE remains in the initial stage with active uniform dissolution and incomplete film coverage, the corrosion current is readily dispersed by widespread uniform dissolution after pit initiation, limiting vertical pit growth. This yields the many-but-shallow pitting morphology in **Figure 4(b)**.

4.3 XPS Analysis of Corrosion Product Films

Figure 9 shows the XPS survey spectra and high-resolution $\text{Cu}2p$, $\text{Ni}2p$, $\text{Fe}2p$, and $\text{Al}2p$ spectra of the corrosion films on the CR

and HE specimens after 14 d of immersion in 3.50 wt% NaCl solution. The $\text{C}1s$ peak at 284.80 eV was used for charge correction. The survey spectra indicate that $\text{Cu}2p$, $\text{Ni}2p$, and $\text{Al}2p$ peaks are relatively strong in both specimens, whereas $\text{Fe}2p$ and $\text{Mn}2p$ signals are weak, and the $\text{Cl}2p$ signal of the HE specimen is more intense than that of the CR specimen. The high-resolution spectra were fitted with Advantage software, Version 5.9 (Thermo Fisher Scientific, Waltham, MA, USA), and peak deconvolution was performed according to standard binding energy references. The results are detailed below.

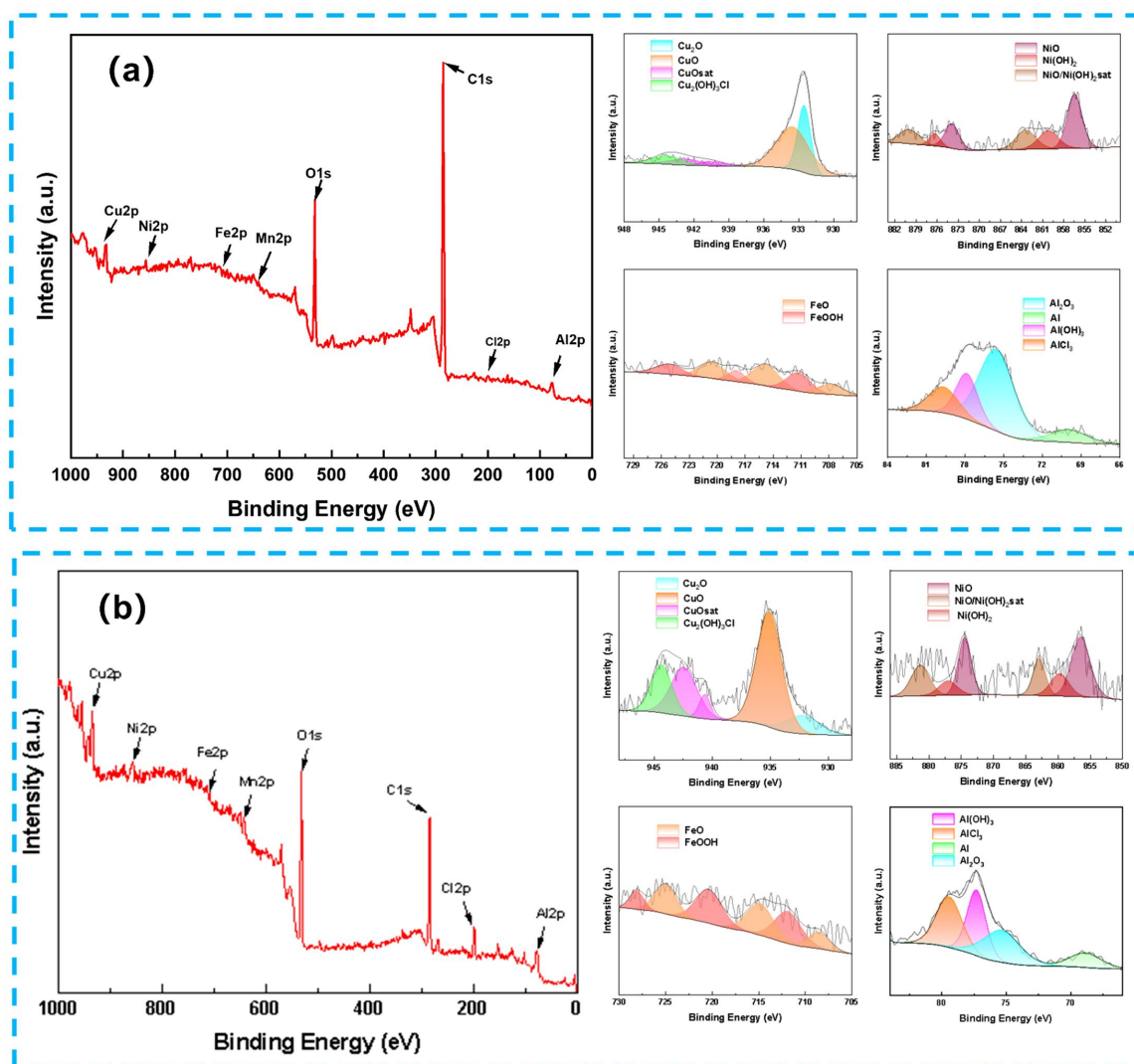


Figure 9. XPS spectra of CR and HE specimens after 14 d of immersion in 3.50 wt% NaCl solution. (a) Survey and high-resolution spectra of CR; (b) Survey and high-resolution spectra of HE. Copyright: Origin 2021, PowerPoint 2016.

The $\text{Cu}2p$ high-resolution spectra show markedly different peak shapes between the two specimens: the CR specimen exhibits a peak shape resembling standard $\text{Cu}^+(\text{Cu}_2\text{O})$, while the HE specimen shows a peak shape similar to standard $\text{Cu}^{2+}(\text{CuO})$, indicating a difference in the $\text{Cu}^+/\text{Cu}^{2+}$ ratio. Following fitting, both specimens present two component peaks at the main peak position, each being a mixture of Cu^+ and Cu^{2+} , with Cu^+ assigned to Cu_2O and Cu^{2+} to $\text{Cu}_2(\text{OH})_3\text{Cl}$ and CuO .

For the $\text{Ni}2p$ spectra, given the inherent complexity of the 2p spectra of transition metals^[39], fitting reveals that Ni is predominantly in the Ni^{2+} state. The peak shape resembles that of NiO but with a slight shift, suggesting the coexistence of $\text{Ni}(\text{OH})_2$ with NiO .

The $\text{Fe}2p$ spectra, characterized by complex multiplet splitting and satellite peaks due to the high-spin nature of FeO ^[40], were fitted with reference to standard binding energies and found to correspond to FeOOH and FeO .

The $\text{Al}2p$ spectra display broader and split main peaks compared with the standard. Fitting confirms the coexistence of metallic Al and Al^{3+} , with the latter assigned to Al_2O_3 , $\text{Al}(\text{OH})_3$, and AlCl_3 .

Figure 10 presents the relative contents of various species in the corrosion products on the CR and HE specimens after 14 d of immersion in 3.50 wt% NaCl solution, as determined by XPS analysis. The most pronounced differences between the CR and HE specimens lie in the Cu and Al containing products.

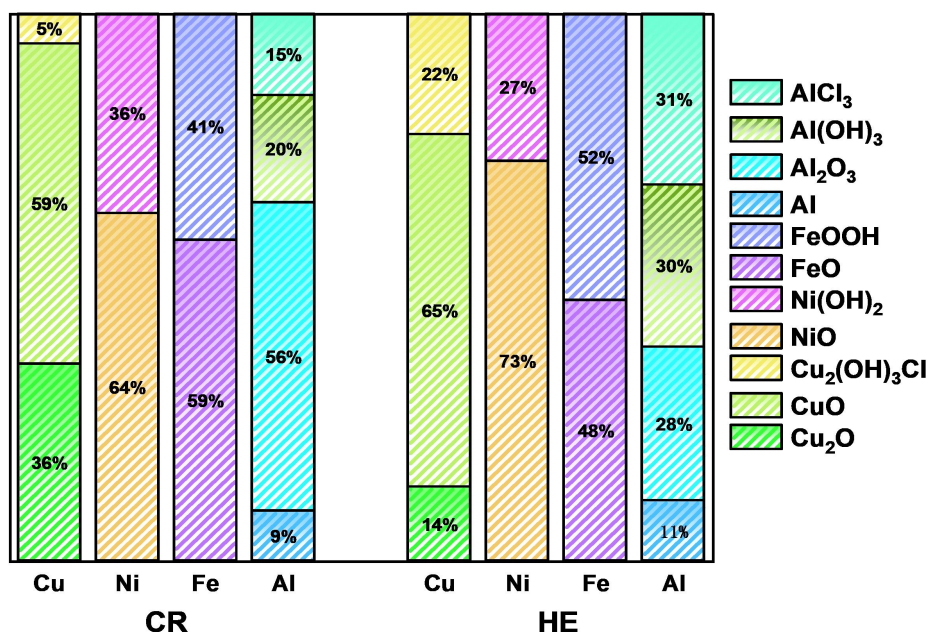


Figure 10. Relative contents of various species in the corrosion products on CR and HE specimens after 14 d of immersion in 3.50 wt% NaCl solution, as determined by XPS analysis. Copyright: Origin 2021, PowerPoint 2016.

In the CR specimen, Cu₂O accounts for 36% of the Cu species, higher than 14% in HE, indicating more Cu₂O retention in CR. CuO is comparable in both states (59% for CR, 65% for HE) and is the predominant component. Cu₂(OH)₃Cl is significantly higher in HE (22%) than in CR (5%), suggesting more extensive transformation from Cu₂O to basic copper chloride in HE.

For Al-containing products, Al₂O₃ accounts for 56% in CR, twice that in HE (28%). In HE, Al(OH)₃ and AlCl₃ are 30% and 31%, respectively, both substantially higher than in CR (Al(OH)₃: 15%). This indicates different Al involvement: in CR, Al tends to form stable Al₂O₃, favoring a dense inner passive film; in HE, Al is predominantly present as Al(OH)₃ and AlCl₃. The higher AlCl₃ proportion in HE implies that Cl⁻ has penetrated the outer corrosion layer to the Al₂O₃/Al(OH)₃ interface and combined with Al³⁺, likely due to the incomplete corrosion film on HE, allowing more severe Cl⁻ penetration.

4.4 In-Depth Analysis of Comparable Corrosion Rates between CR and HE

Despite nearly identical corrosion rates after 14 d, CR and HE exhibit fundamentally different corrosion modes and Al involvement. XPS shows that CR has higher Cu₂O and Al₂O₃, a thicker film, and Al mainly as stable Al₂O₃, which fills defects and enhances the Cl⁻ barrier. HE has more Cu₂(OH)₃Cl, Al(OH)₃, and AlCl₃; the high AlCl₃ indicates Cl⁻ penetration to the Al₂O₃/Al(OH)₃ interface, reflecting an immature film. Nevertheless, HE shows the highest charge transfer resistance (3057 Ω·cm²) and the lowest corrosion current density (1.24 × 10⁻⁵ A/cm²), indicating that its superior corrosion resistance originates from intrinsic protection by fine grains and dense low-Σ CSL boundaries rather than from corrosion products. This highlights the priority of grain boundary engineering over film modification.

In terms of corrosion stage, CR has entered the stable stage with complete film coverage and a low uniform dissolution rate, whereas HE remains in the initial stage with incomplete coverage, exposed substrate, and a high uniform dissolution rate. In terms of

corrosion mode, CR mass loss results from low-rate uniform dissolution plus relatively severe intergranular corrosion, while HE mass loss is dominated by relatively high-rate uniform dissolution with negligible intergranular corrosion. At 14 d, total mass loss is comparable. However, since intergranular corrosion is the primary cause of catastrophic failure, HE is far superior for engineering safety. With prolonged immersion, the HE film will mature, the uniform dissolution rate will decrease, and its corrosion rate will further drop below that of CR, amplifying its intergranular corrosion resistance advantage.

Cross rolling improves compositional and microstructural homogeneity, promotes rapid and complete film coverage, and helps isolate the substrate and reduce pit initiation. However, full recrystallization substantially reduces the absolute populations of low-angle and low-Σ CSL boundaries, leaving a HAGBs-dominated network that allows continuous intergranular corrosion and makes pit propagation difficult to suppress. Hot extrusion, though less effective in homogenization, retains high densities of low-angle and low-Σ CSL boundaries. Although film growth is slower, the dense low-Σ CSL network effectively inhibits intergranular corrosion and pit propagation.

5. Conclusions

(1) The fundamental microstructural difference between CR and HE arises from their distinct recrystallization behaviors. CR, processed by multi-pass small-reduction deformation, achieves sufficient static recrystallization (83.80%) with a homogeneous microstructure and an HAGBs-dominated random grain boundary network. In contrast, HE, subjected to single-pass large-reduction deformation, undergoes incomplete dynamic recrystallization (25%), resulting in a bimodal grain size distribution, a substantial retained deformed structure (47.20%), and a high density of LAGBs.

(2) The absolute density, rather than the relative fraction, of low-Σ CSL grain boundaries is the key parameter governing intergranular corrosion resistance. In CR, despite a relatively high

CSL fraction, the absolute population is limited due to coarse grains, and the interconnected HAGBs network leads to continuous intergranular corrosion. In HE, although the CSL fraction is lower, the extremely high total grain boundary density yields a larger absolute population and denser spatial distribution of CSL boundaries, forming CSL-related triple junctions that effectively block intergranular corrosion and promote uniform dissolution and shallow pitting.

(3) The intrinsic corrosion resistance governed by microstructure takes precedence over the exogenous protection of the corrosion film. Processing routes affect film development by modulating recrystallization, thereby determining whether Al participates mainly as Al_2O_3 or as $\text{Al}(\text{OH})_3/\text{AlCl}_3$. Although the HE film is not fully developed within 14 d, its fine-grained structure and dense low- Σ CSL network provide the highest intrinsic corrosion resistance. In contrast, CR promotes rapid film formation but sacrifices the absolute population of low- Σ CSL boundaries, leaving it unable to effectively suppress intergranular corrosion and pitting, thus compromising long-term stability.

Author Contributions

Conceptualization, Lanqing Niu and Haitao Liu; methodology, Lanqing Niu and Haitao Liu; validation, Lanqing Niu; formal analysis, Lanqing Niu; investigation, Lanqing Niu; resources, Haitao Liu and Kexing Song; writing — original draft preparation, Lanqing Niu; writing — review and editing, Haitao Liu and Kexing Song; supervision, Haitao Liu and Kexing Song; project administration, Haitao Liu. All authors have read and agreed to the published version of the manuscript. Each author has made substantive contributions to the conception, design, or data acquisition, analysis, and interpretation of the study, and has participated in drafting or critically revising the manuscript. All authors have approved the final version and agree to be accountable for all aspects of the work.

Data Availability

The data supporting the findings of this study are available from the corresponding author upon reasonable request.

Conflicts of Interest

The authors declare no competing financial interests.

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