

Article

Corrosion and Mechanical Properties of Inconel 625 Alloy with Gradient Twin Structure Regulated via Interface Engineering

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Abstract

In this paper, solution-treated Inconel 625 alloy was selected as the research object. Combined with the low-energy characteristic of annealing twin boundaries and the synergistic strengthening effect of gradient structures, the alloy microstructure was tailored without altering its chemical composition. The evolution mechanism of grain size and twin boundary fraction, as well as their influence on corrosion resistance, were investigated. On this basis, the synergistic strengthening effect of gradient structure and interface regulation on the corrosion and mechanical performance of the alloy was further explored. The results show that a gradient-structured Inconel 625 alloy with abundant annealing twins and high-density grain boundaries can be fabricated by surface nanocrystallization combined with high-temperature short-time annealing. The variation in annealing twin fraction with grain size in interface-modified Inconel 625 complies with the Pande model. In terms of corrosion resistance, the microstructure consisting of fine grains and a high fraction of annealing twins exhibits superior performance compared to coarse grains with identical twin content. Its corrosion potential is tripled, while the corrosion current density is reduced by 11 times, delivering outstanding anti-corrosion capability in NaCl solution. Benefiting from the integrated merits of gradient architecture, annealing twins and fine grains, the interface-tailored Inconel 625 alloy achieves excellent comprehensive performance, with a yield strength of 925 MPa, elongation of 26%, and corrosion current density of 3.02×10^{-8} A/cm², realizing the integration of high strength, good ductility and superior corrosion resistance for Inconel 625 alloy.

Keywords: Inconel 625 alloy; gradient structure; interface regulation; corrosion performance; mechanical properties



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1. Introduction

Inconel 625 alloy is designed with abundant Cr addition primarily to guarantee corrosion resistance. However, when exposed to Cl[−]-containing environments, rapid surface corrosion occurs while insufficient Cr diffusion from the alloy matrix fails to sustain passive film formation, creating vulnerable corrosion zones and eventually triggering material failure. Notably, the high-density grain boundaries within the nanograined (NG) surface layer of gradient structures provide fast diffusion channels for Cr atoms, enabling rapid replenishment of Cr to the alloy surface and facilitating the growth of compact Cr-rich passive films to block subsequent corrosion propagation [1,2]. As the matrix constituent of

Inconel 625, Ni further stabilizes the structural integrity and ductility of the passive film, suppressing local rupture and pit expansion. Meanwhile, existing studies have verified that the corrosion failure of face-centered cubic (FCC) metals in diverse corrosive media is closely correlated with grain boundary microstructure [3]. As low-energy grain boundaries, annealing twin boundaries ($\Sigma 3$ boundaries) possess remarkable corrosion resistance, and coherent annealing twins are barely corroded at all [4–6]. Thermomechanical processing (TMP) is commonly adopted to modulate the fraction of annealing twins in low-stacking-fault-energy FCC alloys, optimizing grain boundary character distribution (GBCD) and significantly elevating corrosion resistance [7].

Previous investigations have proven that the introduction of gradient structures effectively enhances the mechanical properties of metallic materials [8,9]. By integrating the advantages of gradient microstructure and interface engineering, high-temperature short-time annealing is employed to generate abundant annealing twins on the surface of gradient-structured Inconel 625 while preserving the gradient distribution of grains from the surface to the core. This strategy further improves mechanical properties on the premise of retaining outstanding corrosion resistance, which serves as a critical approach to expand the service scope of Inconel 625 in complex corrosive environments. Accordingly, this work adopts a “nanocrystallization + interface regulation” strategy. Scanning electron microscopy (SEM), electron backscatter diffraction (EBSD), X-ray photoelectron spectroscopy (XPS), and electrochemical corrosion tests are utilized to reveal the corrosion mechanism of alloys with distinct microstructures. Combined with gradient structural design, a high-performance Inconel 625 alloy integrating strength, ductility, and corrosion resistance is targeted, laying a theoretical foundation for broader applications of wrought nickel-based superalloys.

2. Materials and Methods

2.1. Materials

The widely applied solid-solution-strengthened wrought nickel-based superalloy Inconel 625 was selected as the experimental material. Table 1 presents its measured bulk chemical composition of the final fabricated alloy. Several rod-shaped specimens with dimensions of $\Phi 15 \times 110$ mm were cut from the central zone of hot-extruded Inconel 625 alloy bars via wire-electrode discharge machining. Subsequently, these rod specimens were solution-annealed at 1150 °C for 1 h, followed by air cooling (AC). After heat treatment, uniform equiaxed austenite grains and a small amount of annealing twins were formed inside the specimens, and the average grain size was measured to be 92 ± 4 μm .

Table 1. Main chemical composition of Inconel 625 alloy (wt%).

Ni	Cr	Nb	Mo	C	Fe	Si	S	Al	Ti	P
Bal.	21.77	3.75	8.79	0.042	3.68	0.12	0.0006	0.21	0.40	0.006

2.2. Methods

Surface mechanical grinding treatment (SMGT) was performed to construct gradient structures on Inconel 625 surfaces. Prior to the SMGT, a 1 mm-thick surface layer of the specimen was removed via turning to exclude the detrimental impacts of surface oxidation, surface impurities, and coaxiality errors on subsequent processing. During SMGT, the specimen was driven by the chuck and rotated at 1000 rpm. Meanwhile, a hemispherical WC-Co tool tip with a diameter of 8 mm was indented into the specimen surface to a penetration depth of 20 μm . After indentation, the tool tip traveled along the transverse direction of the specimen surface at a constant velocity of 120 mm/min. One pass was

finished when the tool tip slid from one end to the other end of the alloy rod, and 10 passes were applied in this experiment. Oil cooling was adopted throughout the entire processing procedure. The SMGT-processed alloy specimens were subjected to isothermal annealing at 1150 °C in a tube furnace for holding durations of 1 min, 3 min, 5 min, and 10 min, respectively. Prior to annealing, the alloy samples were hermetically sealed in argon-filled quartz tubes for atmosphere protection, and titanium sponge was placed inside each tube to eliminate oxygen contamination. Before loading the sealed specimens, the tube furnace was preheated to 1150 °C and held for 30 min to stabilize the furnace temperature. After the preset holding time was completed, the quartz tubes were taken out rapidly and quenched in water.

A CHI660F potentiostat (CH Instruments, Shanghai, China) was utilized for electrochemical corrosion characterization, including open-circuit potential (OCP), electrochemical impedance spectroscopy (EIS), and potentiodynamic polarization measurements. Bulk specimens with dimensions of $3 \times 5 \times 3$ mm were sectioned from the surface layer of machined and heat-treated Inconel 625 alloy bars by wire-electrode discharge machining for corrosion tests. The wire-cutting marks on samples were polished off using abrasive sandpapers, followed by soldering wires to the back surface of each specimen. To avoid interference with corrosion results caused by corrosion on non-test surfaces and soldered areas, all surfaces excluding the SMGT-treated testing surface were encapsulated with epoxy resin, ensuring the soldered wires were isolated from the corrosive medium. A standard three-electrode electrolytic cell was adopted, with a platinum sheet ($20 \times 20 \times 0.1$ mm) as counter electrode, saturated calomel electrode (SCE) as reference electrode, and the alloy specimen as working electrode with an exposed area of 0.15 cm^2 . Fresh electrolyte and working electrodes were replaced for each test. The corrosive medium was 3.5 wt% NaCl (AR, Sinopharm Chemical Reagent Co., Ltd., Shanghai, China) aqueous solution tested at a constant room temperature of 20 °C. Before formal testing, cathodic polarization at -1.0 V was applied for 200 s to remove native surface oxide films, followed by 1200 s OCP stabilization. EIS tests were conducted under stabilized OCP with an AC perturbation amplitude of 5 mV and a frequency range of 0.01 Hz–100 kHz. Potentiodynamic polarization curves were scanned from -1.0 V to 1.0 V at a scanning rate of 5 mV/s. The sample preparation procedures and gradient-structure characterizations have been described in detail in our previously published articles [10].

2.3. Microstructure and Property Characterization

The preparation flowchart of SEM cross-sectional samples is shown in Figure 1, where the white zone represents the gradient layer, the blue zone denotes coarse grains, and the green zone stands for electrodeposited nickel coating (dimensions in the schematic are not to scale). A FEI Verios 460 scanning electron microscope (SEM, FEI Company, Hillsboro, OR, USA) with an accelerating voltage of 15 kV and probe current of 1.6 nA was used to characterize the cross-sectional microstructures of differently treated Inconel 625 specimens. Backscattered electron (BSE) mode was adopted to distinguish crystallographic orientation differences. Electron backscatter diffraction (EBSD) equipped on a FEI Nano SEM Nova 430 was applied to quantify grain sizes and interfacial orientation relationships across different gradient regions, with an accelerating voltage of 20 kV, probe current of 6.0 nA, working distance of 9.8 mm, and scanning step size ranging from $0.05 \text{ }\mu\text{m}$ to $0.5 \text{ }\mu\text{m}$ adjusted according to local grain dimensions.

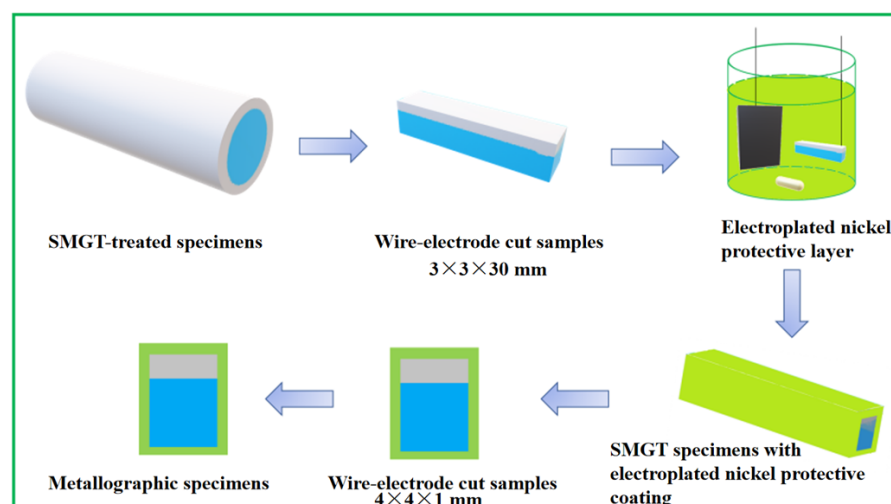


Figure 1. Schematic diagram for cross-section sample preparation for SEM observation.

A Thermo Fisher ESCALAB XI + X-ray photoelectron spectrometer (XPS) equipped with Al K α radiation (1486.6 eV) was employed to characterize corrosion products and elemental valence states formed on alloy surfaces after electrochemical tests. The C 1s peak at 284.8 eV was adopted as the calibration standard; Casa XPS (Version 2.3, Casa Software Ltd., Wilmslow, UK) software was used for peak deconvolution of full survey spectra and high-resolution elemental spectra based on standard binding energy databases. Uniaxial tensile tests were performed on miniature sheet specimens using a Zwick Z2.5 TH universal tensile machine (ZwickRoell GmbH & Co. KG, Ulm, Germany) with a 2.5 kN load cell and laserXtens non-contact extensometer. All tensile measurements were conducted at room temperature with a strain rate of $1 \times 10^{-3} \text{ s}^{-1}$. Defective and flawed specimens were repeatedly eliminated throughout all experimental procedures. Outlying data points were excluded, and only consistent datasets obtained under identical experimental conditions were adopted for subsequent analysis and discussion.

3. Results and Discussion

3.1. Surface Microstructure of Gradient Twin-Structured Inconel 625

For alloys with medium and low stacking fault energy, high-temperature annealing after heavy plastic strain facilitates the gradual nucleation and growth of recrystallized grains. The annealing twins generated in this process also expand progressively with grain boundary migration, by which the fraction of annealing twin boundaries can be tailored. SMGT introduces gradient microstructures into solution-treated Inconel 625, as illustrated in Figure 2a–c, which display the homogeneous austenitic matrix of the as-solutionized alloy and its gradient-modified counterpart. The initial gradient layer thickness of the alloy is approximately 600 μm . After short-time high-temperature annealing at 1150 $^{\circ}\text{C}$ (1–10 min), the alloy surface is dominated by fine recrystallized grains densely populated with annealing twins, alongside a small quantity of abnormally coarsened recrystallized grains (Figure 2d–g). Although obvious recrystallization occurs within the gradient surface layer, the grain size gradient from surface to matrix is well preserved, achieving the target of abundant twin generation while retaining gradient architecture. Meanwhile, the thickness of the gradient layer after heat treatment remains above 300 μm .

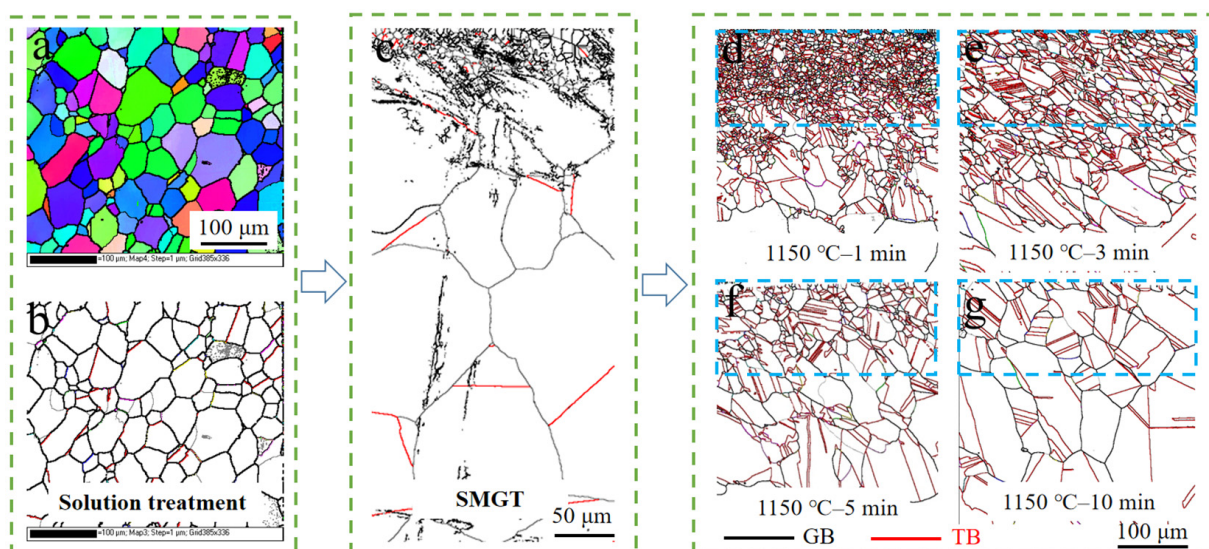


Figure 2. EBSD maps of Inconel 625 under different microstructural states: (a,b) IPF and OIM maps of solution-treated alloy; (c) OIM map of gradient-structured alloy surface; (d–g) OIM maps of interface-modified gradient alloy.

Since alloy corrosion resistance is primarily governed by surface microstructure, grain size and annealing twin fractions of the surface layer after 1150 °C annealing were statistically analyzed within the blue rectangular regions marked in Figure 2d–g, with statistical results presented in Figures 3 and 4. Figure 3 reveals that the fraction of annealing twin boundaries rises gradually with prolonged annealing time, exhibiting a fast initial growth rate followed by slow deceleration. At early annealing stages (1 and 3 min), twin fractions rapidly reach 32% and 45%. For conventional wrought coarse-grained Inconel 625 treated by traditional thermomechanical grain boundary engineering (GBCD), the baseline twin fraction of as-received rolled-solution alloy is merely 13.8% [11]. Even after prolonged high-temperature annealing for 60–90 min, the maximum fraction of annealing twin boundaries only reaches 36% in interface-modified coarse-grained Inconel 625 alloy [4,12]. In contrast, the gradient-structured surface layer fabricated by SMGT attains a high annealing twin fraction of 45% after only 3 min of annealing at 1150 °C, which outperforms all reported counterparts under equivalent short-duration thermal treatment. The rapid increase in twin boundary fraction stems from the unique growth kinetics of annealing twins and the pre-strained gradient nanostructure introduced by SMGT. The severely deformed surface layer contains abundant dislocation substructures, which provide adequate nucleation sites for annealing twins. These twins can grow autonomously without requiring the long-distance migration of random high-angle grain boundaries [13]. In addition, twin boundaries exhibit much lower interfacial energy than conventional high-angle grain boundaries. During annealing, the system reduces its free energy by replacing high-energy random grain boundaries with low-energy twin boundaries, thereby promoting the rapid accumulation of twin boundaries [14]. Unlike conventional long-term high-temperature annealing, which inevitably causes severe grain coarsening and the precipitation of brittle intermetallic phases, the short-time annealing strategy adopted in this work achieves a high twin boundary density while effectively preserving fine surface grains.

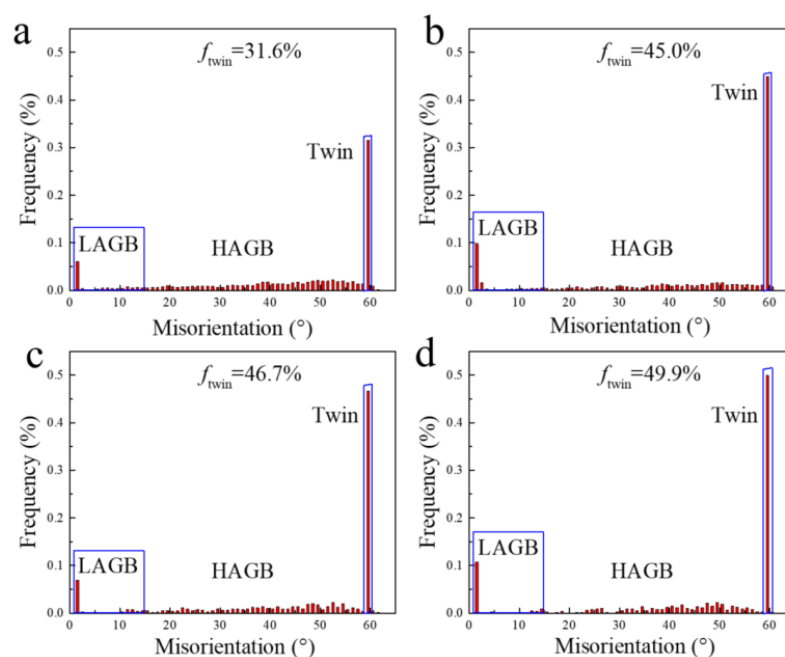


Figure 3. Misorientation distribution statistics of gradient-structured Inconel 625 after different annealing durations: (a) 1 min; (b) 3 min; (c) 5 min; (d) 10 min.

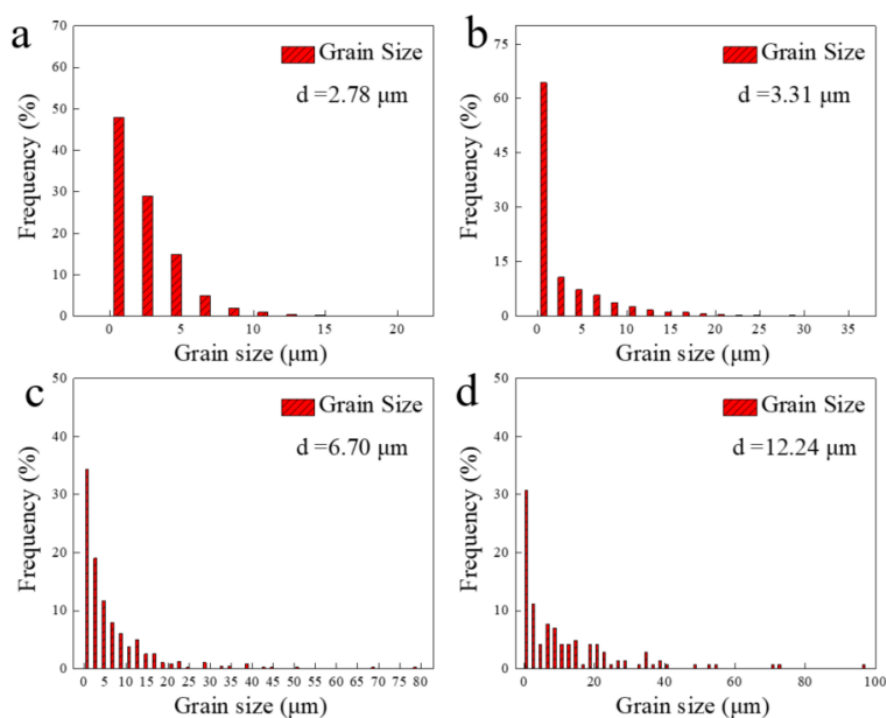


Figure 4. Grain size distribution statistics of gradient-structured Inconel 625 after different annealing durations: (a) 1 min; (b) 3 min; (c) 5 min; (d) 10 min.

Nevertheless, the growth rate of twin fractions slows significantly with further annealing, to 46.7% (5 min) and 49.9% (10 min), representing mere 4% and 11% increments compared with the 3 min condition. As revealed in Figure 4, the average surface grain size of the Inconel 625 alloy rises by 19% (3 min), 141% (5 min) and 340% (10 min) with the extension of annealing time. This trend is completely opposite to the variation in annealing twin fraction. As the grain size further increases, the growth rate of annealing twin fraction gradually slows down. A similar phenomenon was reported by Li et al. [15] during the annealing process of Ni-Fe-based wrought superalloys. Specifically, the twin boundary

fraction increases sharply in the fine-grain size range, while it remains nearly constant with the continuous coarsening of grains. It can thus be concluded that the evolution of annealing twin fraction in Inconel 625 is strongly correlated with grain size.

3.2. Interface Regulation Mechanism of Gradient Structures

Prolonged annealing after plastic nanocrystallization induces prominent grain coarsening, a thermodynamic process that reduces total interfacial area and evolves low-energy boundaries to lower system free energy. Due to the ultra-low interfacial energy of annealing twins, grain growth proceeds primarily via reverse migration of high-angle grain boundaries (HAGBs) against their curvature, as schematically demonstrated in Figure 5. During annealing and HAGB migration, twin T1 gradually shrinks and may annihilate, while twin T2 continuously expands. Meanwhile, atomic misarrangements induced by grain boundary migration generate stacking faults, which further propagate to form new annealing twins such as T3 [16]. The competitive generation and annihilation of annealing twins fundamentally govern the variation trend of twin fractions. Derivation based on the growth accident model proposed by Gleiter [17] indicates that twin fraction at a fixed annealing temperature is uniquely determined by average grain size.

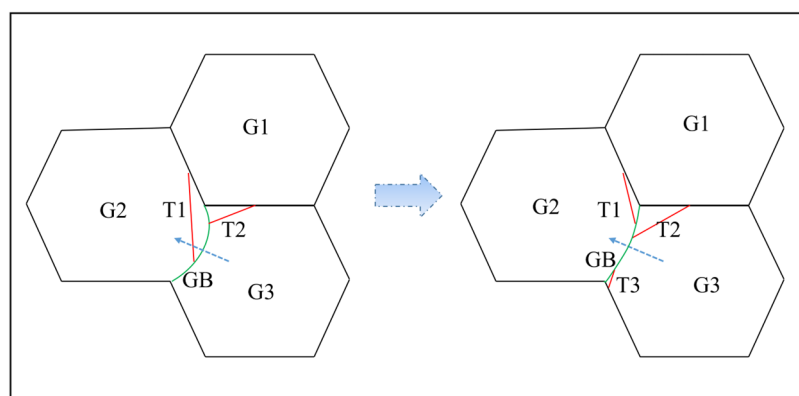


Figure 5. Schematic diagram of grain growth and twin boundary annihilation (G1, G2, G3: three individual grains; T1, T2, T3: annealing twin boundaries; GB: ordinary high-angle grain boundary; blue arrows: grain boundary migration direction).

According to Pande's research on thermomechanically processed FCC alloys [18], the density of annealing twin boundaries is predominantly controlled by grain size, with annealing temperature exerting minor influence. Pande simplified Gleiter's twin growth accident model and derived the formula for twin boundary density ρ :

$$\rho = \frac{C\delta}{d} \ln\left(\frac{d}{d_0}\right)$$

where C denotes a material-specific constant, δ is interfacial energy with unit of $\text{J}\cdot\text{m}^{-2}$, d represents average grain size with unit of μm , and d_0 stands for the critical grain size for twin nucleation, which is fixed for a given alloy and independent of external processing parameters [18,19]. The Pande model curve in Figure 6 illustrates that twin density rises rapidly with grain enlargement within fine-grain domains yet declines continuously for coarse grains, which exhibits consistency with the experimental results obtained in this work and serves as one piece of supporting evidence for the present finding. The relationship between annealing twin fraction and grain size in interface-tailored Inconel 625 agrees reasonably well with the prediction of the Pande model, though certain deviations are expected for this complex multi-component superalloy.

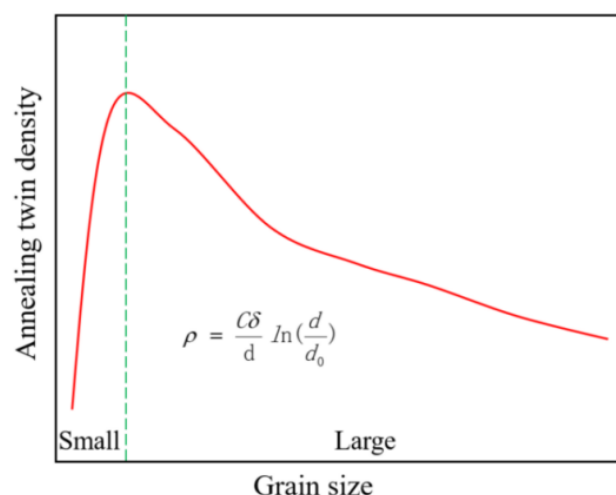


Figure 6. Influence of grain size on twin boundary density (Pande model).

In summary, short-time high-temperature annealing (1–10 min) is adopted in this study to avoid excessive surface grain coarsening, which would eliminate the gradient grain-size distribution and degrade mechanical performance. This process successfully fabricates target microstructures featuring high twin fractions and refined grains.

3.3. Electrochemical Corrosion Behavior of Interface-Regulated Gradient Inconel 625 Alloy

3.3.1. Potentiodynamic Polarization Curves

Gradient-structured Inconel 625 with abundant annealing twins was characterized via electrochemical corrosion measurements. Potentiodynamic polarization tests in 3.5 wt% NaCl solution were carried out for specimens subjected to different processing schedules, and the results are presented in Figure 7 and Table 2. As annealing time extends from 1 min to 10 min, the corrosion potential E_{corr} gradually varies from -0.314 V to -0.833 V, i.e., changes by 0.519 V in the more active direction. Meanwhile, the corrosion current density i_{corr} increases from 3.02×10^{-8} A/cm² to 3.25×10^{-7} A/cm², corresponding to a 10.8-fold increase. Contrary to initial expectations, elevated twin fractions accompanied by grain coarsening degrade alloy corrosion resistance, which is analyzed in the following discussion.

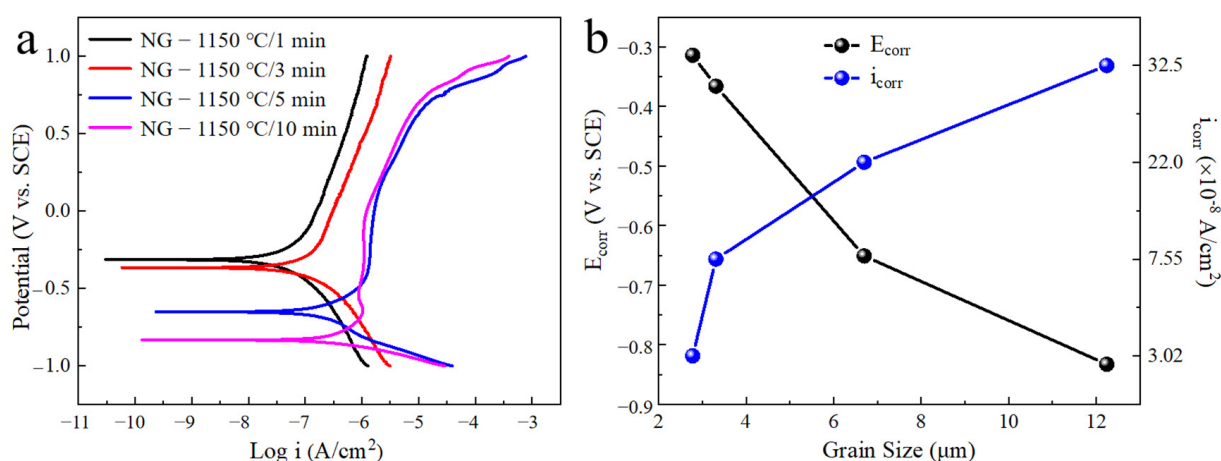


Figure 7. Corrosion behavior of Inconel 625 annealed for different durations in 3.5 wt% NaCl solution: (a) Potentiodynamic polarization curves; (b) Variation in E_{corr} and i_{corr} with grain size.

Table 2. Electrochemical parameters fitted from polarization curves of Inconel 625 alloy subjected to different treatments in 3.5 wt.% NaCl solution.

	E_{corr} (V vs. SCE)	i_{corr} (A/cm ²)
NG–1150 °C/1 min	−0.314	3.02×10^{-8}
NG–1150 °C/3 min	−0.366	7.55×10^{-8}
NG–1150 °C/5 min	−0.651	2.20×10^{-7}
NG–1150 °C/10 min	−0.833	3.25×10^{-7}

Although twin fractions increase with prolonged annealing, concurrent grain coarsening plays a dominant role in corrosion performance. Nanograined (NG) microstructures possess extremely high grain boundary density, which homogenizes free energy and potential differences between grains and boundaries, suppressing localized corrosion initiation [20]. High-density grain boundaries also accelerate the formation of compact passive films by providing active electron sites [21]. The fast Cr diffusion channel effect of fine-grained high-boundary microstructures further improves corrosion resistance. However, long-term annealing induces dramatic grain coarsening, diminishing the corrosion optimization effect originating from abundant grain boundaries. Ordinary high-angle grain boundaries are high-energy crystalline defects with elevated free energy and electronic potential relative to grain interiors, establishing micro-galvanic cells that accelerate interfacial corrosion [22]. Coarse-grained (CG) alloys suffer more severe heterogeneous corrosion due to larger potential discrepancies at grain boundaries. As demonstrated in Figure 7b, the deterioration of corrosion resistance with extended annealing originates mainly from grain coarsening. Microstructures with refined grains and abundant annealing twins deliver a threefold higher corrosion potential and an order-of-magnitude lower corrosion current density compared with coarse-grained samples containing identical twin fractions, verifying their superior corrosion resistance in NaCl solution. Such synergistic fine-grain and high-twin microstructural features offer remarkable anticorrosion advantages via grain boundary interface tailoring under the present testing conditions.

Furthermore, the absence of localized micro-electrochemical testing and three-dimensional grain boundary reconstruction constitutes a clear limitation of this study. Additional corrosion experiments simulating harsher practical service environments will be conducted in follow-up work to fully assess the long-term anticorrosion durability of the gradient microstructure developed herein.

3.3.2. Electrochemical Impedance Spectroscopy

EIS measurements were performed to characterize the impedance response of Inconel 625 with different annealing treatments in NaCl media, as shown in Figure 8. All Nyquist plots display depressed incomplete capacitive semicircles whose radii shrink progressively with prolonged annealing and grain coarsening, verifying deteriorated corrosion resistance. This phenomenon arises from reduced grain boundary density in coarse microstructures, slowing passive film formation kinetics. As summarized in Table 3, both the gradient passive film resistance R_2 and charge transfer resistance R_3 of the alloy increase first and then decrease with prolonged annealing time. Larger values of charge transfer resistance and film resistance correspond to higher resistance to interfacial electrochemical reactions and a slower penetration rate of corrosive media, which signifies superior corrosion resistance. Combined with the foregoing microstructure characterization results, the population of low-energy interfaces rises rapidly at the early annealing stage followed by a slow growth, while the grain size undergoes a fast initial increase and then slow coarsening. It can thus be concluded that the rapid growth of low-energy interfaces at the early annealing stage contributes to improved corrosion resistance. In contrast, the drastic enlargement

of grain size in the later stage leads to a remarkable deterioration in corrosion resistance. The corresponding Bode plots (Figure 8b) reveal that low-frequency impedance declines by up to 2.5 times with extended annealing, while high-frequency impedance remains nearly constant, representing the electrolyte resistance plateau. Two characteristic phase angle peaks appear across the frequency spectrum; the high-frequency peak corresponds to double-layer capacitance, and the low-frequency peak is associated with surface passive film capacitance. Both phase angles are lower than -80° , with the low-frequency region approaching -90° over a wider frequency range, indicating superior protective performance of passive films formed on fine-grained specimens.

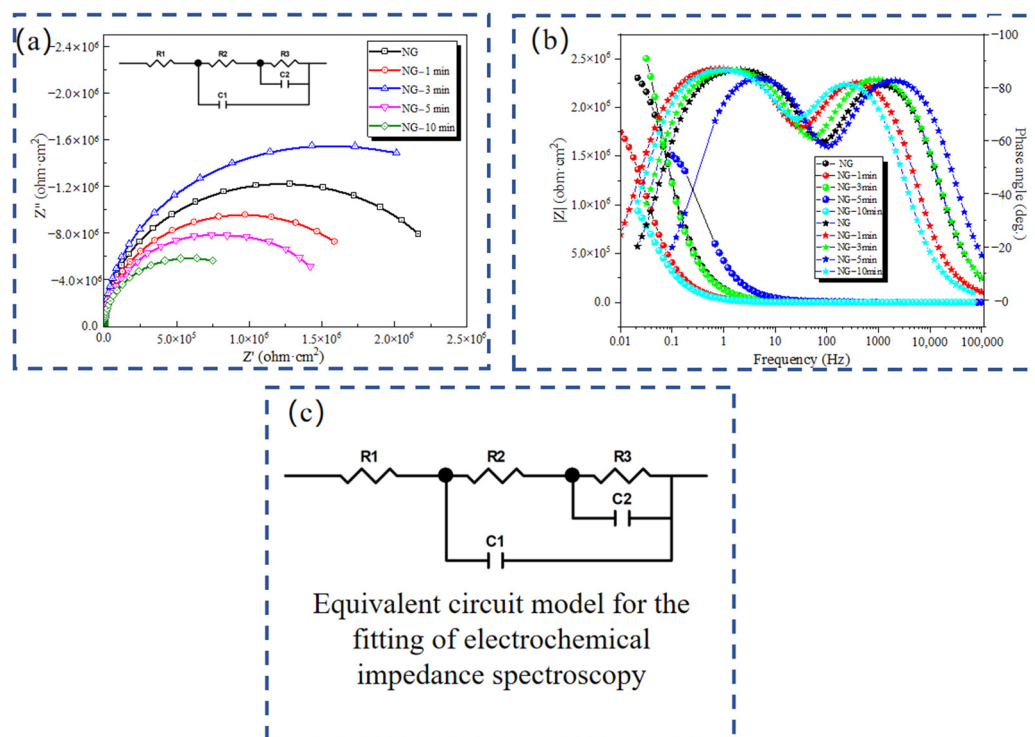


Figure 8. EIS spectra of Inconel 625 with different treatments in 3.5 wt% NaCl solution: (a) Nyquist plots; (b) Bode plots; (c) Equivalent circuit model (electrolyte resistance R_1 , gradient passive film resistance R_2 , charge transfer resistance R_3 , and ideal capacitors C_1 and C_2).

Table 3. Fitted EIS equivalent circuit parameters of Inconel 625 alloy.

Sample	R_1 ($\Omega \cdot \text{cm}^2$)	R_2 ($\Omega \cdot \text{cm}^2$)	C_1 ($\text{F} \cdot \text{cm}^{-2}$)	R_3 ($\Omega \cdot \text{cm}^2$)	C_2 ($\text{F} \cdot \text{cm}^{-2}$)
NG	29.9	4712	3.7×10^{-7}	2.5×10^6	7.4×10^{-7}
NG-1 min	19.1	3617	1.6×10^{-6}	1.9×10^6	2.2×10^{-6}
NG-3 min	25.4	6785	4.2×10^{-7}	3.1×10^6	7.7×10^{-7}
NG-5 min	46.9	11,072	1.1×10^{-7}	0.2×10^6	2.5×10^{-7}
NG-10 min	21.8	3608	2.2×10^{-6}	1.2×10^6	2.5×10^{-6}

3.3.3. Corrosion Morphology and Product Characterization

Corrosion morphologies of differently structured Inconel 625 after immersion in NaCl solution are displayed in Figure 9. Nanocrystallized and interface-modified specimens exhibit uniform surface wrinkling without obvious localized corrosion. In contrast, CG samples show severe intergranular corrosion and discrete corrosion pits with diameters of 3–5 μm distributed along grain boundaries and precipitates. Pitting corrosion is effectively suppressed in nanograined microstructures, consistent with electrochemical data.

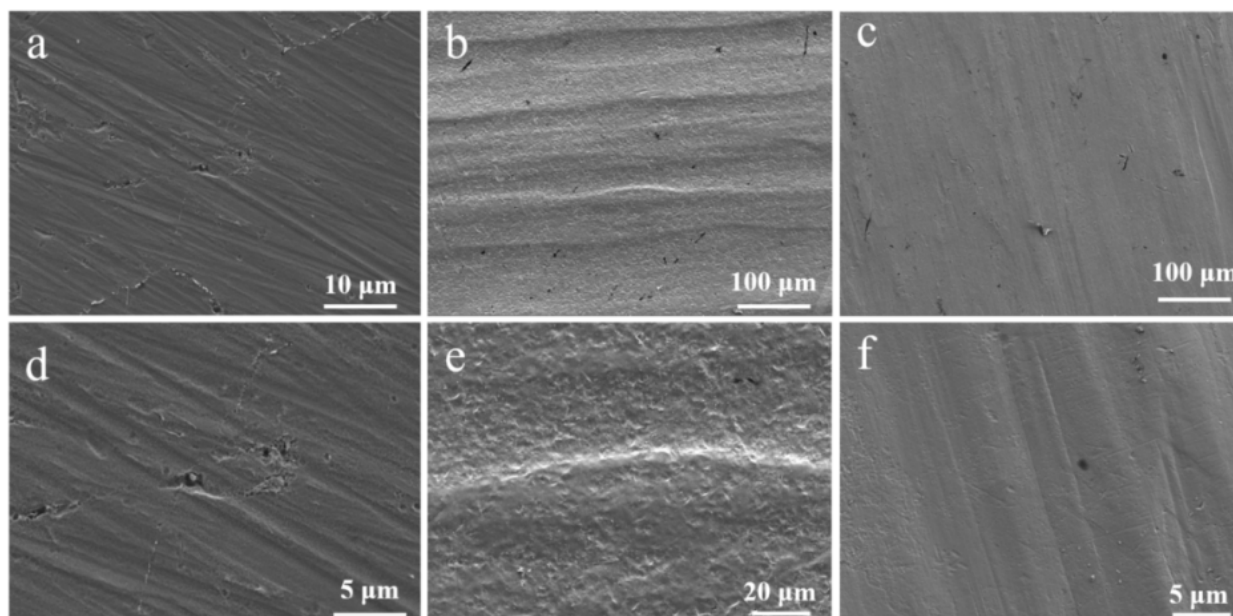


Figure 9. Corrosion morphologies of Inconel 625 with distinct microstructures after immersion in 3.5 wt% NaCl solution: (a,d) Coarse-grained alloy; (b,e) Nanograined alloy; (c,f) Nanocrystallized + interface-modified alloy.

Figure 10 presents XPS full survey and high-resolution spectra of corrosion products formed on interface-regulated gradient alloy surfaces. The full spectrum (Figure 10a) confirms the presence of Ni 2p, Cr 2p, and O 1s signals after corrosion. High-resolution XPS spectra (Figure 10b–d) correspond to the characteristic peaks of Ni 2p_{3/2}, O 1s, Cr 2p_{3/2} and Cr 2p_{1/2}, respectively. For spectral deconvolution, background signals were eliminated via the Shirley baseline correction method, and mixed Gaussian–Lorentzian line shapes were utilized to resolve overlapping characteristic peaks. By comparing the relative integrated peak area ratios of respective chemical species derived from Figure 10b–d, obvious differences in the constituent composition of corrosion product layers can be observed between different specimens. Three distinct chemical states of nickel were detected on the corroded alloy surface, namely metallic Ni at 852.7 eV, nickel oxide (NiO) at 855.8 eV, and nickel hydroxide (Ni(OH)₂) at 861.6 eV. Chromium mainly forms chromium sesquioxide Cr₂O₃ at 576.4 eV and chromium hydroxide Cr(OH)₃ with binding energies of 577.4 eV and 586.8 eV. The oxygen signals are assigned to NiO (529.8 eV) and Ni(OH)₂ (531.7 eV). XPS analysis demonstrates that the corrosion products formed on the alloy surface after corrosion consist of metallic oxides (NiO, Cr₂O₃) and metallic hydroxides (Ni(OH)₂, Cr(OH)₃), which are basically consistent with the surface products of nanograined (NG) alloys after corrosion. The protective Cr₂O₃ and NiO oxide layers are responsible for the corrosion resistance of the alloy. Within the present experimental conditions (room temperature, 3.5 wt% NaCl solution, and fixed immersion duration), the applied microstructure tuning strategy does not induce obvious variations in the chemical composition of surface corrosion products. The enhanced corrosion resistance originates from two aspects: one is low-energy annealing twin boundaries with intrinsic excellent corrosion resistance; the other is abundant grain boundaries introduced by grain refinement, which accelerate Cr diffusion and homogenize the surface potential distribution, thereby improving the overall corrosion resistance of the alloy.

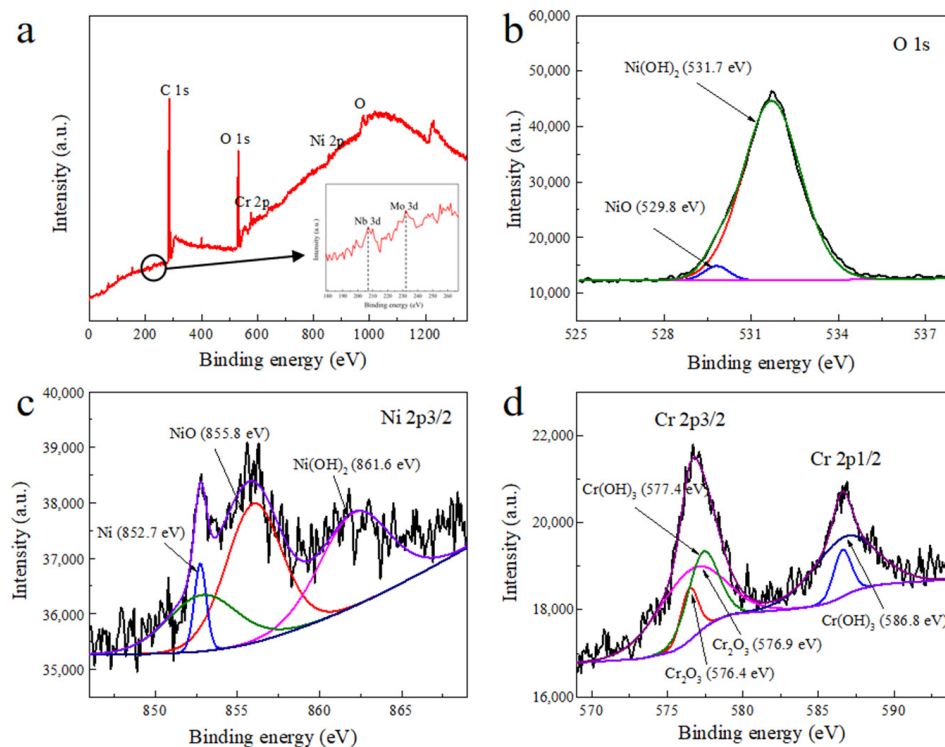


Figure 10. XPS spectra of corroded interface-modified gradient Inconel 625 in 3.5 wt% NaCl solution: (a) Full survey spectrum; (b–d) High-resolution spectra of individual elements.

3.3.4. Mechanical Properties of Interface-Regulated Gradient Inconel 625

Interface tailoring of grain size and twin fractions significantly improves the corrosion resistance of Inconel 625 while retaining the gradient architecture responsible for outstanding mechanical performance. Tensile test results are summarized in Figure 11 and Table 4. Nanocrystallized and interface-modified alloys exhibit far higher strength (925 MPa) than CG counterparts. After 1 min annealing, yield strength only decreases by 11% yet remains 2.7 times that of the as-solutionized CG alloy, with an elongation of 26%. For commercial solid-solution-treated wrought Inconel 625 alloy, the standard yield strength is only 460 MPa with an elongation of 50% [23]. Traditional grain boundary engineering (GBCE) on coarse-grained Inconel 625 can raise the twin fraction to 36%, yet its yield strength merely reaches ~520 MPa at an elongation of 27%, showing obvious strength loss at equivalent ductility [4]. Additively manufactured SLM Inconel 625 exhibits a maximum yield strength of 554 MPa and elongation of 45.3% [24]. With prolonged annealing from 1 min to 10 min, yield strength gradually declines from 925 MPa to 399 MPa, while ductility increases from 26% to 53%. The high strength of gradient alloys originates from the synergistic strengthening of surface nanograins and gradient strain partitioning. When annealing exceeds 5 min, severe surface grain coarsening weakens grain-boundary strengthening, and the gradient microstructure gradually homogenizes, drastically reducing the synergistic hardening effect and bringing strength close to the original alloy level.

As shown in Figure 12 and Table 5, interface regulation via SMGT combined with short-time high-temperature annealing successfully fabricates Inconel 625 alloy with fine grains, high twin fractions, and preserved gradient distribution. The optimized alloy achieves an excellent comprehensive performance, exhibiting a yield strength of 925 MPa, an elongation of 26%, and a low corrosion current density of 3.02×10^{-8} A/cm².

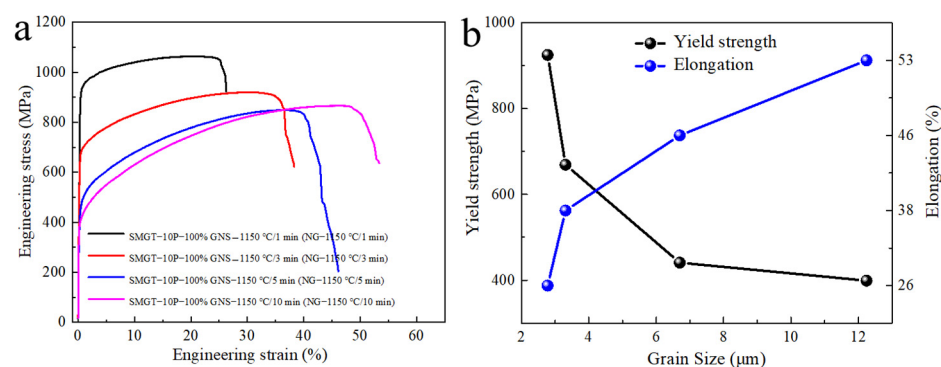


Figure 11. Tensile properties of Inconel 625 under different processing routes: (a) curves; (b) Correlation between yield strength, elongation, and grain size.

Table 4. Mechanical properties of Inconel 625 alloys prepared via different processes.

Sample	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation (%)
NG-1150 °C/1 min	925	1064	26
NG-1150 °C/3 min	669	920	38
NG-1150 °C/5 min	441	849	46
NG-1150 °C/10 min	399	866	53

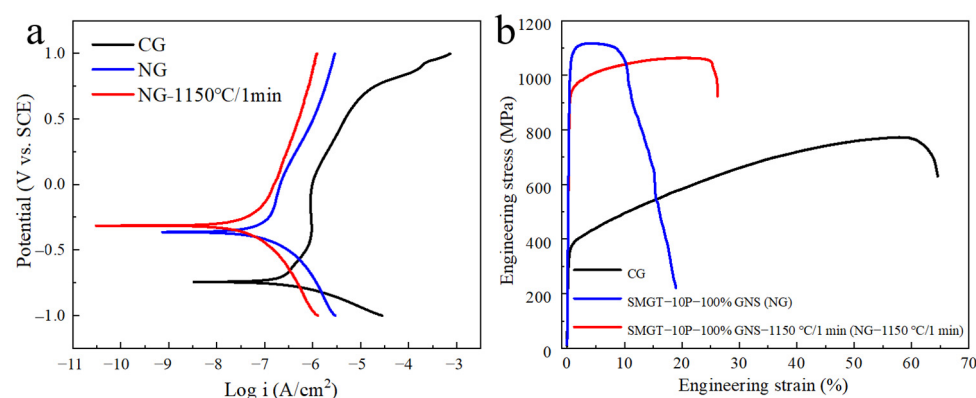


Figure 12. (a) Corrosion and (b) mechanical performance of Inconel with different fabrication processes.

Table 5. Mechanical and corrosion properties of Inconel 625 alloys under different processes.

Sample	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation (%)	E_{corr} (V vs. SCE)	i_{corr} (A/cm ²)
CG	342	773	65	−0.744	3.33×10^{-7}
NG	1040	1117	19	−0.364	7.55×10^{-8}
NG-1150 °C/1 min	925	1064	26	−0.314	3.02×10^{-8}

Interface regulation by SMGT coupled with annealing treatment enables the fabrication of Inconel 625 alloy with refined grains, a high fraction of annealing twin boundaries, and retained gradient structure, as displayed in Figure 2. Performance characterizations reveal that the alloy modified by such interface regulation exhibits outstanding comprehensive mechanical properties and corrosion resistance, with a yield strength of 925 MPa, an elongation of 26%, and a self-corrosion current density of 3.02×10^{-8} A/cm², as summarized in Figure 12 and Table 4.

Previous research indicated that pure nanograined alloys with high internal strain energy and defect densities are susceptible to pitting at grain or phase boundaries [25].

However, SMGT-processed Inconel 625 achieves uniform elemental distribution and minimal secondary phase precipitation on the gradient surface, reducing interfacial potential discrepancies and suppressing localized corrosion. Similar observations have been reported for pure Fe, Fe-based and Ni-based alloys [26,27]. As-fabricated gradient nanograined Inconel 625 already combines favorable mechanical properties and corrosion resistance (Figure 13). Further short-time high-temperature annealing introduces abundant low-energy annealing twins, releases residual strain, and reduces defect density to homogenize elemental distribution, moderately improving ductility with only a minor sacrifice of strength, realizing comprehensive performance optimization. The overall performance tuning flowchart is illustrated in Figure 13.

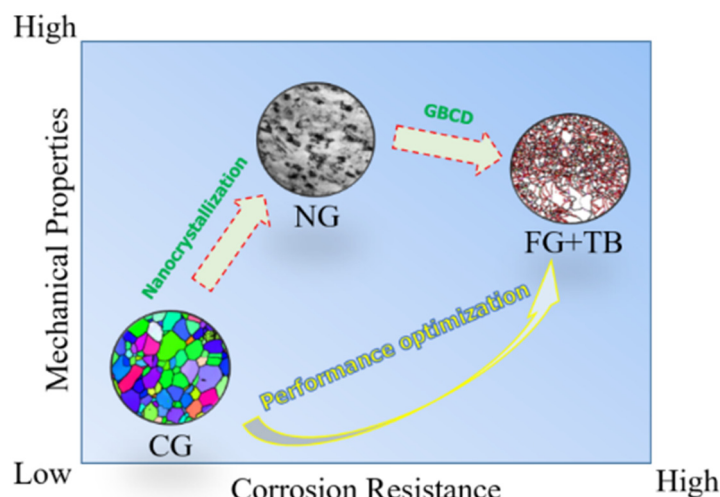


Figure 13. Schematic diagram of performance optimization for high-strength corrosion-resistant gradient Inconel alloy (CG: coarse grain; NG: nanograin; FG: fine grain; TB: twin boundary).

4. Conclusions

Based on the successful fabrication of a gradient-structured Inconel 625 alloy with superior mechanical properties, surface microstructure was further optimized via interface engineering. SEM, EBSD, XPS, and electrochemical measurements were adopted to characterize the mechanical and corrosion behavior of alloys with distinct surface microstructures. The main conclusions are summarized as follows:

Surface nanocrystallization (SMGT) coupled with high-temperature short-time annealing at 1150 °C for 1–10 min successfully produces a gradient-structured Inconel 625 alloy with abundant annealing twins and high-density grain boundaries. The dependence of annealing twin fraction on grain size follows the Pande model. After 1 min and 3 min annealing, surface twin fractions reach 32% and 45%, surpassing the maximum twin content of interface-modified coarse-grained solutionized Inconel 625.

The corrosion resistance of twin-containing gradient Inconel 625 varies significantly with annealing duration. The corrosion potential E_{corr} decreases gradually from -0.314 V to -0.833 V, while the corrosion current density i_{corr} rises from 3.02×10^{-8} A/cm² to 3.25×10^{-7} A/cm² as annealing time extends. Specimens with fine grains and high twin fractions (short annealing) exhibit a tripled corrosion potential and an 11-fold lower corrosion current relative to CG alloys with identical twin content, demonstrating superior resistance to chloride corrosion. Microstructures consisting of fine grains and abundant annealing twins are optimal for corrosion protection during interface regulation.

Benefiting from the synergistic advantages of gradient architecture, fine grains, and abundant annealing twin boundaries, the interface-regulated gradient-structured Inconel 625 alloy exhibits excellent mechanical properties and corrosion resistance. It possesses

a yield strength of 925 MPa and an elongation of 26%, along with a low self-corrosion current density of 3.02×10^{-8} A/cm². This efficient microstructure tuning strategy enables balanced improvements in room-temperature mechanical properties (strength and ductility) and NaCl corrosion resistance of Inconel 625 alloy under the tested experimental conditions.

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Conflicts of Interest: Author Zhou Chen was employed by the company Lanzhou Wanli Aero-Electromechanics Co., Ltd. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

References

1. Wang, L.; Li, D.Y.; Lu, K. Effect of surface nanocrystallization on friction and wear properties of low carbon steel. *Mater. Sci. Eng. A* **2003**, *352*, 144–149. [\[CrossRef\]](#)
2. He, Q.; Wei, W.; Wang, M.S. Gradient Microstructure Design in Stainless Steel: A Strategy for Uniting Strength-Ductility Synergy and Corrosion Resistance. *Nanomaterials* **2021**, *11*, 2356. [\[CrossRef\]](#) [\[PubMed\]](#)
3. Yang, H.; Xia, S.; Zhang, Z.L. Improving the intergranular corrosion resistance of the weld heat-affected zone by grain boundary engineering in 304 austenitic stainless steel. *Acta Metall. Sin.* **2015**, *51*, 333–340.
4. Gao, Y.B.; Zhen, B.; Xu, J.Y. Achieving strength–ductility synergy and enhanced intergranular corrosion resistance in the Inconel 625 alloy via twin boundary tailoring. *J. Mater. Res. Technol.* **2025**, *35*, 3728–3740. [\[CrossRef\]](#)
5. Gao, Y.B.; Ding, Y.T.; Chen, J.J. Grain boundary character distribution and texture evolution during short-process fabrication of GH3625 alloy tubes. *Rare Met. Mater. Eng.* **2020**, *49*, 1996–2003.
6. Gao, Y.B.; Li, X.Y.; Ma, Y.J. Formation mechanism and wear behavior of gradient nanostructured Inconel 625 alloy. *Trans. Nonferrous Met. Soc. China* **2022**, *32*, 1910–1925. [\[CrossRef\]](#)
7. Han, C.; Liu, Y.; Wang, Y. Hot Corrosion behavior of Ni-xCr-6.8Al based alloys. *Trans. Nonferrous Met. Soc. China* **2011**, *21*, 2348–2357. [\[CrossRef\]](#)
8. Guo, F.J.; Wang, Y.F.; Wang, M.S. Hetero-deformation induced strengthening and toughening of pure iron with inverse and multi-gradient structures. *Mater. Sci. Eng. A* **2020**, *782*, 139256. [\[CrossRef\]](#)
9. Lu, K. Gradient nanostructured metals: A new frontier in materials science. *Science* **2014**, *345*, 1455–1456. [\[PubMed\]](#)
10. Ma, Y.J.; Ding, Y.T.; Gao, Y.B. Gradient Nanotwinned Structure and Its Formation Mechanism in Inconel 625 Alloy Produced by Surface Mechanical Grinding Treatment. *Met. Mater. Int.* **2022**, *29*, 1454–1468. [\[CrossRef\]](#)
11. Liu, A.S. Discussion on EBSD Analysis Method for Microstructure of Inconel 625 Alloy. *Met. World* **2021**, 27–30.
12. Ma, Y.J.; Wang, J.J.; Gao, Y.B. Improving Intergranular Corrosion Resistance in Inconel 625 via Grain Boundary Character Distribution Optimization. *J. Wuhan Univ. Technol.-Mater. Sci.* **2021**, *36*, 911–921. [\[CrossRef\]](#)
13. Jin, Z.H.; Gumbsch, P.; Ma, E. The interaction mechanism of screw dislocations with coherent twin boundaries in different face-centred cubic metals. *Scr. Mater.* **2006**, *54*, 1163–1168. [\[CrossRef\]](#)
14. Randle, V. Twinning-related Grain Boundary Engineering. *Acta Mater.* **2004**, *52*, 4067–4081. [\[CrossRef\]](#)

15. Li, Z.G. Evolution and Mechanical Behavior of Annealing Twin Boundaries in a Ni-Fe Wrought Superalloy. Master's Thesis, Shanghai Jiao Tong University, Shanghai, China, 2015.
16. Pande, C.; Rath, B.; Imam, M. Effect of annealing twins on Hall-Petch relation in polycrystalline materials. *Mater. Sci. Eng. A* **2004**, *367*, 171–175. [[CrossRef](#)]
17. Gleiter, H. The formation of annealing twins. *Acta Metall.* **1969**, *17*, 1421–1428. [[CrossRef](#)]
18. Gleiter, H. The mechanism of grain boundary migration. *Acta Metall.* **1969**, *17*, 565–573. [[CrossRef](#)]
19. Nembach, E. *Particle Strengthening of Metals and Alloys*; John Wiley Sons, Inc.: New York, NY, USA, 1997; pp. 1–286.
20. Miyamoto, H.; Harada, K.; Mimaki, T. Corrosion of ultra-fine grained copper fabricated by equal-channel angular pressing. *Corros. Sci.* **2008**, *50*, 1215–1220. [[CrossRef](#)]
21. Tao, S.; Li, D.Y. Nanocrystallization effect on the surface electron work function of copper and its corrosion behaviour. *Philos. Mag. Lett.* **2008**, *88*, 137–144. [[CrossRef](#)]
22. Xu, X.X.; Niu, F.L.; Wang, Y.B. Effective inhibition of the early copper ion burst release with ultra-fine grained copper and single crystal copper for intrauterine device application. *Acta Biomater.* **2012**, *8*, 886–896. [[CrossRef](#)] [[PubMed](#)]
23. Special Metals. *INCONEL® Alloy 625*; ASM Aerospace Specification Metals, Inc.: Pompano Beach, FL, USA, 2024.
24. Ferraresi, R.; Avanzini, A.; Cecchel, S. Microstructural, Mechanical, and Tribological Evolution under Different Heat Treatment Conditions of Inconel 625 Alloy Fabricated by Selective Laser Melting. *Adv. Eng. Mater.* **2022**, *4*, 2100966. [[CrossRef](#)]
25. Gupta, R.K.; Birbilis, N. The influence of nanocrystalline structure and processing route on corrosion of stainless steel: A review. *Corros. Sci.* **2015**, *92*, 1–15. [[CrossRef](#)]
26. Liu, J. Effect of Surface Nanocrystallization on Corrosion Resistance of Nickel-Based 690 Alloy. Master's Thesis, Jiangxi University of Science and Technology, Nanchang, China, 2020.
27. Xu, H. Study on Corrosion Electrochemical Properties of Fe-50Co Bulk Alloys with Different Grain Sizes. Master's Thesis, Shenyang Normal University, Shenyang, China, 2021.

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