

PREPARATION AND SYNERGISTIC STRENGTHENING MECHANISM OF MULTI - SCALE HETEROGENEOUS 7075 ALUMINUM ALLOY

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This study constructed a heterogeneous structure in 7075 aluminum alloy via friction stir processing (FSP) and aging (150 °C, 20 h). Microstructure and mechanical properties were characterized. FSP produced a gradient structure comprising fine-grained (4–5 μm), transition, and coarse-grained (30–100 μm) zones. Aging precipitated uniform nano-scale second-phase particles, inhibiting grain growth and increasing geometrically necessary dislocation density by ~25%. Nanoindentation showed enhanced deformation resistance across all zones, most markedly in the base material. This synergy between FSP and aging optimizes the performance of aluminum alloy heterogeneous structures.

Keywords: 7075 aluminum alloy; Heterogeneous structure; Friction stir processing (FSP); Aging treatment; Microstructure; Mechanical properties

1. Introduction

As a typical Al-Zn-Mg-Cu ultra-high-strength aluminum alloy, 7075 aluminum alloy is widely used in the main load-bearing structural components of high-end equipment manufacturing fields such as aerospace and transportation due to its specific strength comparable to that of some structural steels, excellent processing performance, and good corrosion resistance [1][2]. However, traditional homogeneous high-strength aluminum alloys generally face the bottleneck problem of the difficulty in synergistically improving strength and plasticity/toughness [3]. This contradiction severely restricts its application potential under extreme working conditions, where both high load-bearing capacity and high damage tolerance are required simultaneously.

To overcome the aforementioned performance bottlenecks, the design concept of “heterostructured materials” inspired by natural biological materials has

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provided a new approach for the simultaneous optimization of the strength and deformation capacity of metallic materials [4][5]. The core idea lies in constructing “soft” (easily deformable) and “hard” (difficult to deform) regions with significant differences in performance or mechanical response within the material. By utilizing the back stress and additional work hardening capacity generated by heterogeneous deformation-induced (HDI) strengthening, the occurrence of necking can be effectively delayed while increasing the strength, thereby improving the uniform plastic deformation capacity of the material [6][7]. Among numerous heterostructure fabrication techniques, Friction Stir Processing (FSP), as an efficient solid-state plastic processing technology, can successfully construct a grain size gradient heterostructure composed of fine-grained zones, transition zones, and base material zones in aluminum alloys through its intense plastic deformation and dynamic recrystallization effects, showing great application prospects [8][9]. However, for heat-treatable aluminum alloys such as 7075, the high-temperature thermal cycle during the FSP process can cause the dissolution of strengthening phases in the stirred zone and overaging in the thermomechanically affected zone, leading to the loss of control over the precipitation state of the as-processed heterostructure, and its strengthening potential is far from being fully exploited [10][11]. The inherent inhomogeneity of the as-processed microstructure and the resulting instability of mechanical properties make it difficult for the simple FSP gradient structure to meet the stringent requirements of high-performance applications. Therefore, subsequent aging heat treatment of the heterostructure fabricated by FSP to precisely control its precipitation behavior and release the strengthening potential is a crucial step in optimizing its comprehensive performance.

Currently, there is still a lack of systematic research on the influence of aging treatment on the microstructure evolution law and performance regulation mechanism of the heterogeneous structure of 7075 aluminum alloy prepared by friction stir processing (FSP). Based on this, this paper takes 7075 aluminum alloy as the research object, prepares the heterogeneous structure using FSP technology, and focuses on studying the influence law of aging treatment on its microstructure evolution and mechanical properties. By systematically analyzing the microstructure characteristics and performance changes of the heterogeneous structure before and after aging treatment, the aim is to clarify the regulation mechanism of aging treatment on the strengthening and toughening of the heterogeneous structure, providing a theoretical basis for optimizing the heat treatment process of high-performance aluminum alloy heterogeneous structures.

2. Experimental section

2.1 Experimental materials

The base material used in the experiment was a commercially rolled 7075

aluminum alloy plate with dimensions of 300 mm (rolling direction, RD) \times 100 mm (transverse direction, TD) \times 10 mm (normal direction, ND). The composition of the material was analyzed using a spark direct-reading spectrometer, and the results are shown in Table 1. Before friction stir processing, the surface of the plate was sanded with sandpaper to remove the oxide layer, then ultrasonically cleaned with alcohol to remove oil stains, and dried with cold air.

Table 1

Element composition and content of 7075 aluminum alloy

Element	Al	Mg	Ti	Zn
Wt (%)	Bal.	2.68	0.21	5.76
Cu	Fe	Cr	Mn	Si
1.54	0.20	0.21	0.06	0.07

2.2 Experimental methods

The rolled sheet was first solution-treated at 475 °C (water quenched) to obtain the T4 state. A TS-1260 friction stir welding machine was used for processing, and the schematic diagram is shown in Fig. 1. The length of the stirring pin was 3 mm, the tilt angle of the stirring head was 1.5°, the rotation direction was clockwise, and the plunge depth was controlled at 0.1 mm. The processing parameters were fixed at a rotational speed of 1000 rpm and a travel speed of 70 mm/min. The processed samples were naturally cooled in the air. Then, specimens were cut from the processed samples, and the schematic diagram of sampling is shown in the figure. Subsequently, the samples were subjected to single-stage aging treatment: 150 °C/20 h. All the aging treatments were carried out in a DZF-6020B vacuum drying oven. After completion, the samples were taken out and air-cooled to room temperature.

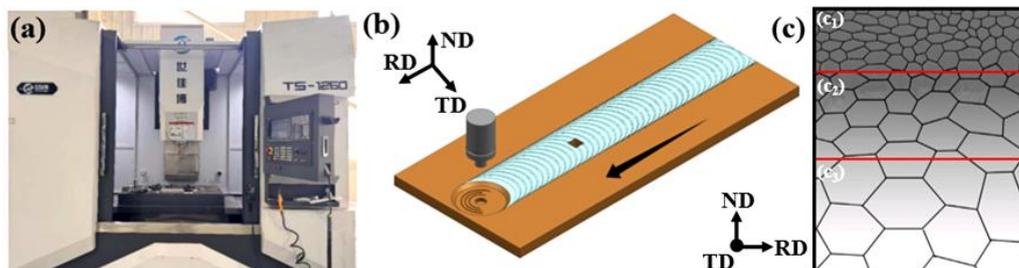


Fig. 1. Schematic diagrams of FSP processing and sampling: (a) TS-1260 friction stir welding machine; (b) schematic of FSP process; (c) sampling locations, where (c1), (c2), and (c3) denote the fine-grained zone, transition zone, and base material zone, respectively.

2.3 Experimental methods

The specimens used for microstructural characterization were all cut

perpendicular to the FSP direction. After cold mounting, grinding, and polishing, the specimens were etched with Keller's reagent (2.5 mL HNO₃ + 1.5 mL HCl + 1.0 mL HF + 95 mL H₂O), and the preliminary microstructural observation was carried out using a Zeiss AxioObserver A1m optical microscope. The morphology and composition of the precipitated phases were analyzed by a Nova Nano 450 field emission scanning electron microscope (SEM) equipped with an energy - dispersive spectrometer (EDS). To prepare for electron back - scatter diffraction (EBSD) analysis, the specimens were vibration - polished with a SiO₂ suspension (180 Hz, ≥12 h) to remove surface stress. The test voltage was 15 kV, and the data were processed by TSL-OIM™ software to obtain information on grain boundaries, dislocations, and recrystallization. Phase identification was completed using an X-ray diffractometer (XRD, scanning range 10°-90°, speed 5°/min). Meanwhile, a HVST - 1000Z microhardness tester (load 0.98 N, holding time 15 s) was used to plot the hardness distribution on the cross-section of the specimens. The hardness and elastic modulus of local regions were evaluated by a Hysitron TI950 TriboIndenter (Berkovich indenter, maximum load 10 mN).

3. Results and discussion

3.1 Microstructural evolution

The microstructure of the base metal zone exhibits typical rolling-state characteristics. The grains are elongated along the rolling direction, presenting a coarse fibrous shape with a size range of 30 - 100 μm. Coarse second-phase particles can be observed both within the grains and at the grain boundaries (Fig. 2a). After friction stir processing (FSP), significant gradient evolution occurs in the material's microstructure. In the processing core area, the strong mechanical stirring and thermo-mechanical coupling induce severe plastic deformation and dynamic recrystallization. The original coarse grains are refined into fine equiaxed grains with a size of approximately 4 - 5 μm, forming a fine-grained zone (Fig. 2b). In the transition zone adjacent to the fine-grained zone (Fig. 2c), due to the weakened thermo-mechanical effect, the microstructure shows inhomogeneous characteristics: the grain size gradient increases, and some grains retain the elongated shape with torsional deformation. The heat-affected zone outside it is mainly affected by the thermal cycle, with little change in grain size; while the base metal zone remains basically unaffected, still maintaining the original coarse fibrous microstructure.

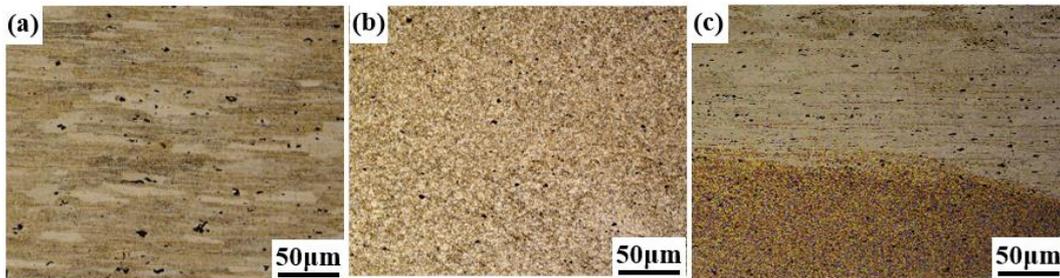


Fig. 2. Optical microstructures of 7075 aluminum alloy under different conditions: (a) T4 state base material zone; (b) FSP fine-grained zone; (c) FSP transition zone.

Fig. 3(a), (d) shows the SEM image of the base metal zone of the T4-state 7075 aluminum alloy. There are many undissolved coarse second-phase particles in the microstructure, which are mainly distributed in the grain boundaries and intra-grain regions. After FSP, the microstructure of the fine-grain zone changes significantly, as shown in Fig. 3(c), (f). The grains in this zone are significantly refined and transformed into a fine equiaxed grain structure. The original coarse second phase is broken and dissolved under the stirring action, accompanied by a re-precipitation process. The fine precipitated phases are relatively uniformly distributed around the grain boundaries, but some undissolved coarse second-phase residues can still be seen at the grain boundaries. Fig. 3(b), (e) shows the microstructure morphology of the transition zone. The grain size in this zone is non-uniform, and some grains still retain certain deformation characteristics. The precipitated phases are discontinuously and densely distributed at the grain boundaries, and the obvious gradient change of the microstructure reflects the transitional characteristics of the thermal - mechanical effect during the friction stir processing.

Analysis shows that although FSP can effectively refine grains and construct heterogeneous structures, the thermal cycle it introduces also causes the dissolution or coarsening of strengthening phases, resulting in the as-processed materials failing to fully realize their precipitation strengthening potential. Therefore, subsequent aging treatment is a key step in regulating the state of precipitated phases and achieving the strengthening and toughening of heterogeneous structures. This subsection will systematically explore the regulatory effects of aging treatment at 150°C for 20 h on the precipitation behavior, grain boundary characteristics, and dislocation configurations of T4 - state FSP samples, aiming to clarify the synergistic strengthening mechanism between aging treatment and the FSP heterogeneous structure. Fig. 2 shows the XRD patterns of T4 - state FSP samples before and after aging treatment at 150°C for 20 h. It can be seen that the α - Al matrix phase mainly exists in the samples. The diffraction peaks corresponding to the (111), (002), (022), (113), and (222) crystal planes are clearly visible, and no obvious new phases are detected, indicating that the aging conditions do not change the phase composition of the materials. Compared with the un - aged samples, the

intensity of the α - Al diffraction peaks changes significantly after aging at 150°C for 20 h, which may be due to the evolution of nano - precipitated phases such as GP zones and η' phases during the aging process.

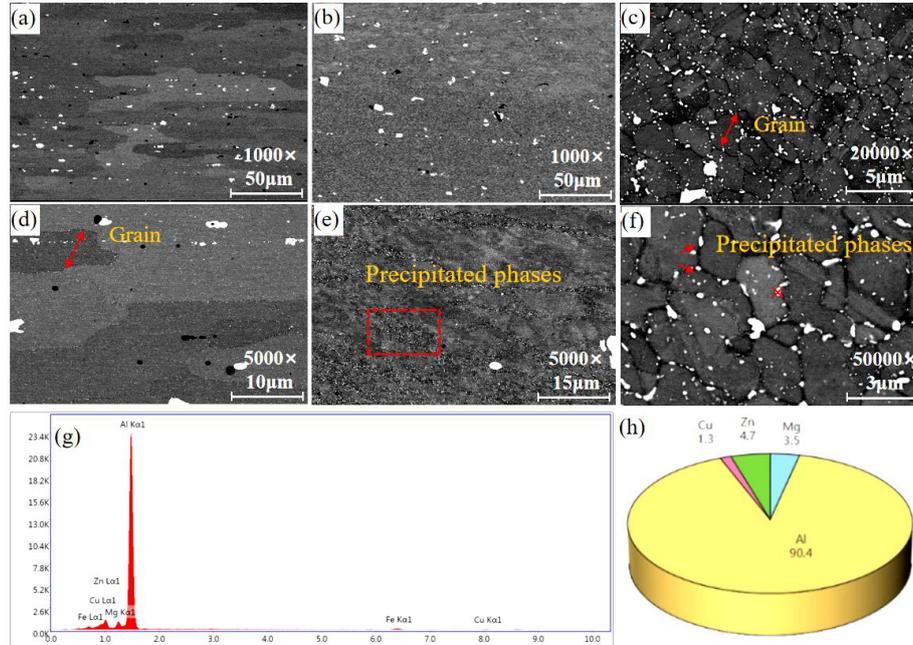


Fig. 3. SEM images of 7075 aluminum alloy before and after FSP: (a), (d) T4 state base material zone; (b), (e) transition zone processed; (c), (f) fine-grained zone processed; (g), (h) the eds of second-phase

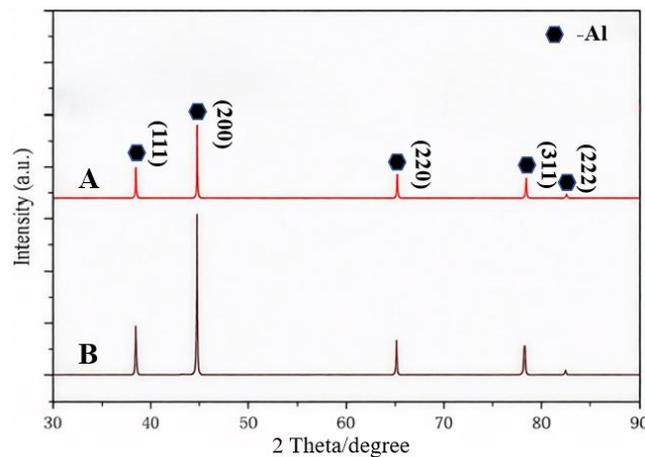


Fig. 4. Phase analysis of 7075 aluminum alloy in T4 state: Sample A-FSP + aging at 150 °C for 20 h; Sample B-FSP only.

The metallographic structure analysis in Fig. 5 (a, b) indicates that after the aging treatment at 150°C for 20 h, both the fine-grained region and the transition

region of the T4 state 7075 aluminum alloy exhibit good structural stability, and no obvious grain coarsening occurs. Meanwhile, the analysis of precipitated phases (Fig. 5c, d) shows that this aging treatment promotes the precipitated phases to appear as fine granular shapes with regular morphologies and relatively uniform size distributions. The large-sized blocky precipitated phases in the original structure basically disappear. These nano-scale precipitates effectively inhibit grain boundary migration through the Zener pinning effect. Together with the high-density grain boundaries formed by FSP, they hinder grain growth. Although the precipitated phases are still unevenly distributed in local areas, overall, the aging treatment at 150°C for 20 h maintains the stability of the fine-grained structure and optimizes the morphology and distribution of the precipitated phases, which helps to maintain the Hall-Petch strengthening effect and dispersion strengthening effect, and is of positive significance for improving the comprehensive properties of the material.

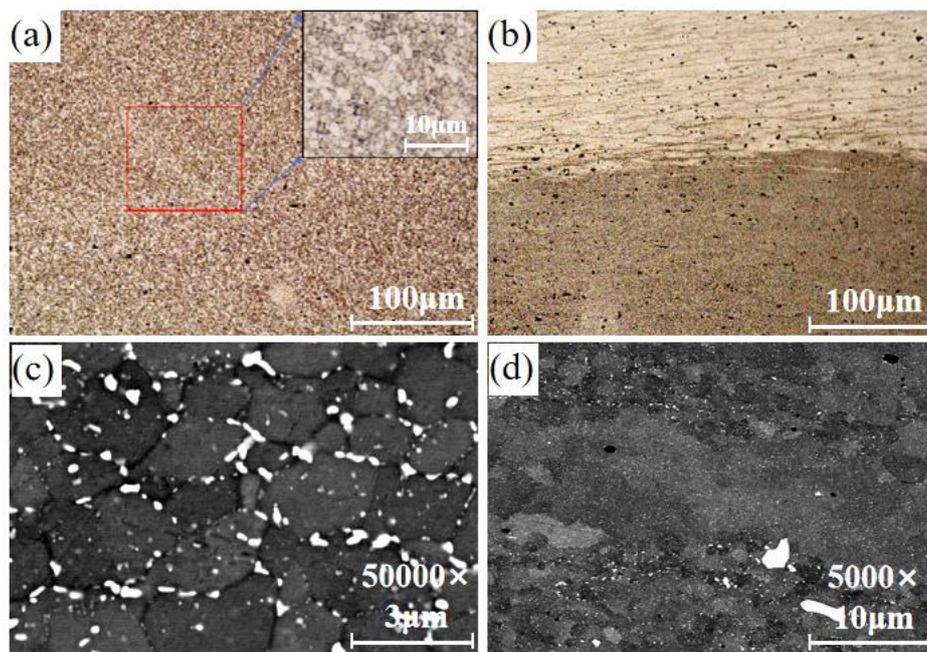


Fig. 5. Microstructural characterization of 7075 aluminum alloy after aging treatment: (a) optical microstructure of FSP fine-grained zone; (b) optical microstructure of FSP transition zone; (c) SEM image of FSP fine-grained zone; (d) SEM image of FSP transition zone.

Fig. 6 (a, d) comparatively shows the recrystallization distribution characteristics of the FSP-state specimen and the specimen after aging treatment at 150 °C for 20 h. The FSP state exhibits a typical dynamic recrystallization structure, with the proportion of recrystallized grains reaching 83.8%, indicating that sufficient recrystallization occurred during the processing. After aging treatment at

150 °C for 20 h, the proportion of recrystallized grains significantly decreased to 55.6%, while the proportions of sub-grains and deformed grains increased to 31% and 13.1%, respectively. This structural transformation indicates that the aging treatment effectively inhibits the grain boundary migration through the pinning effect of the precipitated phase, hindering the further development of the recrystallization process. In terms of grain boundary characteristics, the comparative analysis in Fig. 6 (b, e) shows that the proportion of high-angle grain boundaries (HAGBs) in the FSP specimen is as high as 87.9%. After aging at 150 °C for 20 h, the proportion of HAGBs decreased to 73.9%, and the low-angle grain boundaries (LAGBs) increased accordingly. This change in grain boundary characteristics is in good agreement with the decrease in the degree of recrystallization, further confirming the influence of aging treatment on the structural stability. The decrease in the proportion of HAGBs may affect the crack propagation resistance of the material, but it also enhances the stability of the grain boundaries. Through the analysis of geometrically necessary dislocations (GNDs) (Fig. 6c, f), it is found that the GND density of the FSP specimen is $0.81 \times 10^{14} \text{ m}^{-2}$. After aging treatment at 150 °C for 20 h, the GND density increased to $1.02 \times 10^{14} \text{ m}^{-2}$, with an increase of about 25%. This change indicates that the aging treatment not only affects the grain boundary characteristics but also promotes the redistribution and storage of dislocations. The increase in GND density helps to improve the strain hardening ability of the material and the uniformity of deformation.

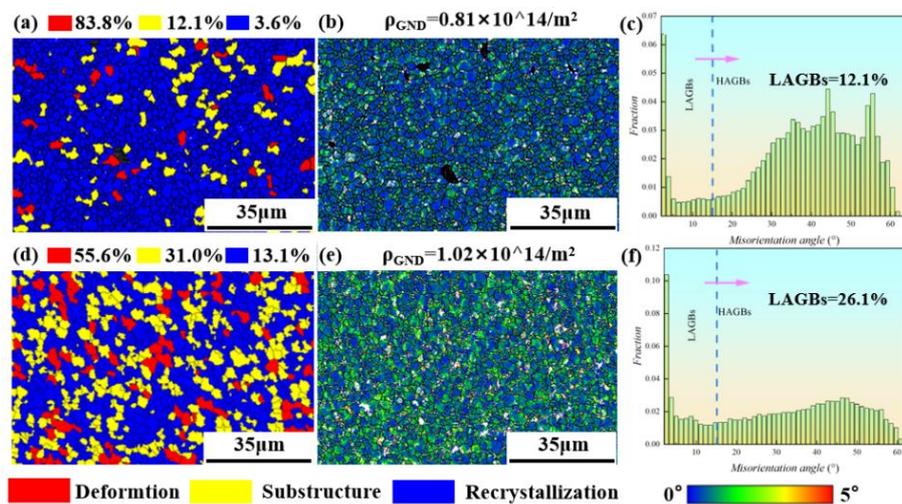


Fig. 6. EBSD characterization results of 7075 aluminum alloy under different conditions: (a-c) fine-grained zone processed by FSP; (d-f) fine-grained zone processed by FSP + aging at 150°C/20h; (a, d) grain orientation spread (GOS) maps; (b, e) geometrically necessary dislocation (GND) density distributions; (c, f) statistical distributions of low-angle and high-angle grain boundaries.

3.2 Nanoindentation

Nanoindentation test results indicate that friction stir processing (FSP) successfully constructs a heterogeneous structure with significant mechanical property gradients in 7075 aluminum alloy. As shown in Fig. 7, the FSP - treated sample exhibits typical characteristics of micro - mechanical property distribution: the fine - grained zone (1 mm from the processed surface) shows the highest nano - hardness and elastic modulus, but its plastic deformation ability is relatively limited; the mechanical property parameters of the transition zone (2 mm and 3.5 mm) are between those of the fine - grained zone and the base metal zone, and the results at different positions show good consistency; the base metal zone (6 mm) demonstrates the optimal plastic deformation ability, but its deformation resistance is the weakest. This continuous change in mechanical properties is closely related to the grain size gradient formed during the FSP process.

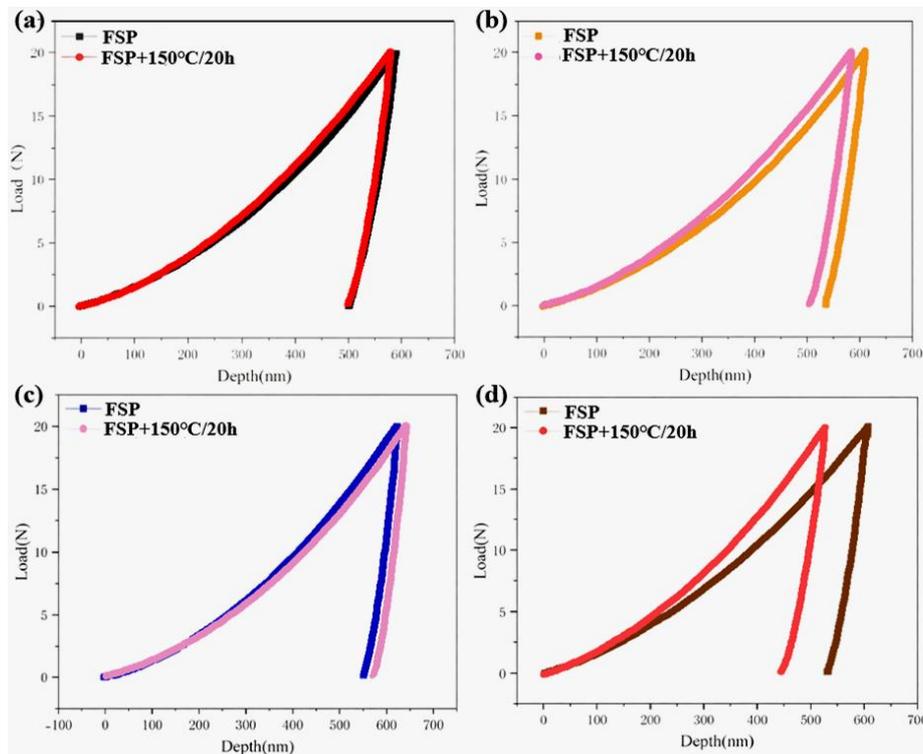


Fig. 7. Nanoindentation load-depth curves of 7075 aluminum alloy across different regions before and after aging treatment.

After aging treatment at 150 °C for 20 h, the mechanical properties of various regions in the material were significantly optimized. On the basis of maintaining their original performance characteristics, the deformation resistance of the fine-grained zone and the transition zone was further enhanced (Figs. 7a - c). Notably, the strengthening effect in the base metal zone was the most remarkable, with its

nano-hardness and deformation resistance being significantly improved (Fig. 7d). This comprehensive improvement in performance is mainly attributed to the nano-scale precipitates formed during the aging process. These precipitates, through an effective dispersion strengthening mechanism, not only enhance the strength of the fine-grained zone and the transition zone but, more importantly, significantly improve the mechanical properties of the base metal zone.

3.3 Strengthening Mechanism

In the T4 - state 7075 aluminum alloy, a heterogeneous structure with multi - scale synergistic strengthening effect was successfully constructed by a combined process of friction stir processing (FSP) and aging at 150 °C for 20 h. The core feature of this structure lies in the composite strengthening system composed of the fine - grained matrix formed by FSP and the high - density nano - phases precipitated during the aging process. The nano - precipitates strongly impede the dislocation movement through the coherent strain field, providing significant precipitation strengthening. Meanwhile, these precipitates effectively pin the grain boundaries, stabilizing the fine - grained structure obtained by FSP and enabling the full play of grain boundary strengthening. More importantly, the heterogeneous structure induces a significant strain gradient during deformation, prompting the large - scale multiplication and storage of geometrically necessary dislocations at the interface, resulting in a strong heterogeneous deformation - induced strengthening effect. This multi - scale synergistic action not only significantly enhances the material strength but also effectively improves the deformation coordination of the material by enhancing the work - hardening ability and maintaining the grain boundary toughness. The research shows that this process path achieves a good match between strength and plasticity by precisely controlling the size and distribution of precipitates and their interactions with grain boundaries and dislocations, providing new design ideas and process basis for the development of high - performance heterogeneous - structured aluminum alloy materials.

4. Conclusions

Through the research on friction stir processing (FSP) and aging treatment of 7075 aluminum alloy, the following conclusions are drawn:

(1) The aging treatment at 150°C for 20 h effectively maintains the gradient structure formed by FSP. The nano-phase inhibits grain growth through Zener pinning, and the geometrically necessary dislocation density increases by 25%.

(2) Aging treatment significantly improved the deformation resistance of each region, and the strengthening effect was most obvious in the base metal region, mainly due to the dispersion strengthening effect of nano-precipitates.

(3) Through its intense thermo-mechanical coupling effect of FSP , successfully constructed a continuous gradient heterogeneous structure in 7075

aluminum alloy, ranging from the fine-grained zone (4-5 μm) and transition zone to the coarse-grained base material zone (30-100 μm). This provides a crucial microstructural foundation for the subsequent aging treatment to achieve multi-scale synergistic strengthening.

(4) The prepared multi-scale heterogeneous structure promotes strain partitioning and back stress strengthening during deformation due to the significant property differences between the fine-grained and coarse-grained zones, synergistically enhancing the strength and plasticity of the material. The uniformly distributed nano-precipitates formed after aging treatment further augment this heterogeneous deformation-induced (HDI) strengthening effect.

(5) The combination of FSP and aging treatment achieves multi-scale synergy of fine-grain strengthening, precipitation strengthening, and heterogeneous deformation-induced strengthening, providing a new approach for the development of high-performance aluminum alloys.

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