Microstructure and Mechanical Properties of Heat-Treated Laser Solid Formed Inconel 718 Superalloy: Postprint

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Abstract

The microstructure and mechanical properties of laser solid formed Inconel 718 alloy subjected to high-temperature homogenization solution treatment, intermediate d aging treatment, and double-stage aging treatment were investigated, and the dislocation configuration of the heat-treated alloy was examined. The results indicate that due to recrystallization behavior during the heat treatment process, the alloy microstructure transformed from the initial columnar grains in the as-deposited state to equiaxed grains. The Laves phase was completely dissolved, while needle-like d phase and g strengthening phase were dispersedly precipitated at grain boundaries and in the g matrix, respectively. The tensile strength, yield strength, elongation, and reduction of area of the heat-treated laser solid formed Inconel 718 alloy all met the forging standard. The interaction between dislocations and g phase involved both dislocation shearing of g phase and dislocation bypassing of g phase; in regions where dislocations bypassed g phase, the dislocation density was higher than in regions where dislocations sheared g phase. Due to the larger d phase size in the heat-treated state compared to forgings, dislocations piled up within the d phase. Strong interactions also existed between carbides and dislocations, which hindered dislocation motion through pinning and dragging effects.

Full Text

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Abstract

This study investigates the microstructure and mechanical properties of laser solid formed (LSF) Inconel 718 alloy subjected to a three-stage heat treatment consisting of high-temperature homogenization solution treatment, intermediate δ-phase aging, and double aging. The dislocation configurations in the heat-treated alloy were also characterized. The results show that due to recrystallization during heat treatment, the microstructure transforms from the initial columnar grains in the as-deposited state to equiaxed grains. The Laves phase is completely dissolved, while needle-like δ-phase and γ strengthening phase precipitate abundantly along grain boundaries and within the γ matrix, respectively. The tensile strength, yield strength, elongation, and reduction of area of the heat-treated LSF Inconel 718 alloy all meet wrought alloy standards. The interactions between dislocations and γ phase occur through two mechanisms: dislocations shearing through γ phase and dislocations bypassing γ phase. In regions where dislocations bypass γ phase, the dislocation density is higher than in regions where dislocations shear through γ phase. Due to the larger δ-phase size in the heat-treated condition compared to wrought material, dislocations tend to pile up at δ-phase. Strong interactions also exist between carbides and dislocations, which hinder dislocation motion through pinning and drag effects.

Keywords: laser solid forming, Inconel 718, microstructure, mechanical property, dislocation configuration

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Introduction

Inconel 718 is a second-phase precipitation-strengthened Ni-Fe based superalloy widely used in aerospace, energy, and power generation applications due to its high strength, oxidation resistance, radiation resistance, and excellent hot workability and weldability. In recent years, with the development of metal additive manufacturing technology, laser solid forming (LSF) has emerged as an important fabrication method for high-performance, complex Inconel 718 alloy components [?]. Unlike conventional casting, the high temperature gradient and rapid cooling rate during LSF molten pool solidification can expand solute solubility limits, refine substructures, and improve microsegregation to some extent [?, ?]. Additionally, the high power density of laser irradiation easily causes melt superheating. Wang et al. [?] reported that melt superheating effectively refines superalloy dendrite spacing and improves γ phase morphology. Kolotukhin et al. [?] also found that melt superheating can control the quantity and morphology of carbide eutectics while increasing carbide-forming element content in the matrix, thereby enhancing alloy stability. Furthermore, the layer-by-layer deposition approach in LSF produces a repeated reheating/annealing effect, resulting in microstructures different from those obtained by conventional processing methods. Blackwell [?] and Lin et al. [?] observed that LSF-produced Inconel 718 alloy exhibits fine, uniform dendritic structures with distinct banding between layers. Zhao et al. [?] and Liu et al. [?] found that LSF Inconel 718 alloy shows epitaxially grown columnar crystals with identical dendrite orientation within the same columnar grain, demonstrating directional solidification characteristics, though adjacent columnar grains have certain misorientations and a single columnar grain can traverse several deposition layers. Moreover, Zhao et al. [?] reported that the primary dendrite arm spacing in LSF Inconel 718 alloy is approximately 5 m, significantly finer than in cast material. However, LSF Inconel 718 alloy still exhibits some microsegregation of alloying elements in interdendritic regions, and stress analysis reveals large, non-uniformly distributed residual stresses. Liu et al. [?] demonstrated that heat treatment of LSF Inconel 718 alloy can not only eliminate microsegregation but also relieve residual thermal stresses, promote recrystallization, and refine grains, thereby improving alloy strength. Although previous work has analyzed the microstructural characteristics and mechanical properties of LSF Inconel 718 alloy, systematic analysis of the specific strengthening and toughening mechanisms is still lacking.

Generally, alloy strengthening mechanisms are manifested through dislocation configurations, which reflect interactions between strengthening phases and dislocations. Since LSF Inconel 718 alloy typically requires appropriate heat treatment to achieve excellent mechanical properties, this work analyzes the microstructural evolution and tensile mechanical properties of heat-treated LSF Inconel 718 alloy, examines the influence of different strengthening phases on fracture mechanisms, and investigates the strengthening mechanisms of various precipitates through dislocation configuration analysis. This study aims to provide an important scientific foundation for optimizing LSF processing parameters and heat treatment regimes for Inconel 718 alloy.

Experimental Methods

The LSF-III laser solid forming system used in this study consists of an RS-850 4 kW CO_2 laser, an LPM-408 four-axis CNC positioning system, a DPSF-1 high-precision automatic powder feeder, and a coaxial powder delivery nozzle. The substrate was 316L stainless steel with dimensions of 140 mm × 60 mm × 6 mm. The powder was spherical Inconel 718 alloy produced by plasma rotating electrode process, with an average particle size of approximately 150 m and chemical composition (wt.%) of: Cr 19.7, Nb 5.1, Fe 18.4, Al 0.65, Ti 1.04, Mo 2.9, C 0.03, Ni balance. Prior to deposition, the powder was vacuum-dried at 120 °C for 4 h to remove adsorbed moisture, and the substrate surface was ground with sandpaper and cleaned with acetone. The target specimen dimensions were 56 mm × 12 mm × 12 mm. The main LSF process parameters were: laser power 2 kW, scanning speed 8 mm/s, powder feed rate 8–12 g/min, shielding gas flow rate 4–8 L/min, laser spot diameter 3 mm, overlap ratio 40%, and layer thickness 0.2–0.3 mm.

A three-stage heat treatment was applied to the as-deposited samples: high-temperature homogenization solution treatment + intermediate δ-phase aging + double aging. The specific parameters were: 1100 °C for 1.5 h, air-cooled + 980 °C for 1 h, air-cooled + 720 °C for 8 h, furnace-cooled to 620 °C, held for 8 h, air-cooled. Room-temperature tensile testing of the heat-treated LSF Inconel

718 alloy specimens was performed using an INSTRON-96 universal testing machine at a loading rate of 1 mm/min. Three parallel specimens were tested for each condition, and results were averaged. Microstructural characterization was conducted using a PMG3 optical microscope (OM) and a VEGA II-LMH scanning electron microscope (SEM). Fracture surfaces of tensile specimens were analyzed by SEM, and dislocation configurations were examined using a Tecnai G2 F30 transmission electron microscope (TEM). Phase composition before and after the three-stage heat treatment was analyzed using an X'Pert MPD Pro X-ray diffractometer (XRD).

2.1 Microstructure

[Figure 1: see original paper] shows the microstructure of as-deposited LSF Inconel 718 alloy. As seen in Fig. 1a, the structure exhibits epitaxially grown columnar dendrites traversing multiple deposition layers, with columnar grain widths of approximately 200 m and strong growth orientation tending toward the deposition direction. The angle between columnar grain growth direction and deposition direction is approximately 30°. Since the substrate used in this work was polycrystalline with random orientation, and the temperature gradient was maximum at the bottom of the molten pool oriented toward the deposition direction, columnar grains growing epitaxially from the substrate underwent competitive growth, with those having orientations closer to the deposition direction eventually dominating. Due to the high solidification rate of laser melting, the primary dendrite arm spacing within columnar crystals is about 5–10 m (Fig. 1b). Additionally, some irregular particles exist in interdendritic regions (Fig. 1c). EDS analysis reveals severe Nb segregation in these particles (Fig. 1d), identifying them as Laves phase.

[Figure 2: see original paper] shows the microstructure and precipitates of LSF Inconel 718 alloy after high-temperature solution treatment, δ-phase aging, and double aging. High-temperature solution treatment can eliminate residual thermal stresses and alloy element segregation in the as-deposited state, completely dissolving the Laves phase. Moreover, residual thermal stresses generated during LSF can induce recrystallization, transforming the columnar grain structure in as-deposited LSF Inconel 718 alloy into equiaxed grains (Fig. 2a). The equiaxed grains are refined with non-uniform sizes of approximately 50–200 m. δ-phase aging promotes precipitation of fine needle-like δ-phase along grain boundaries (Fig. 2b). The δ-phase appears as fine needles 1–2 m in length, precipitating along grain boundaries, though carbides remain after heat treatment. After double aging, γ phase is observed to precipitate dispersively in the γ matrix (Fig. 2c). The γ phase appears as circular discs in front view and ellipsoids in side view, with disc diameters of 20–50 nm and elliptical minor axes of about 10 nm.

[Figure 3: see original paper] shows XRD spectra of LSF Inconel 718 alloy in as-deposited and heat-treated conditions. Only diffraction peaks from the γ-phase matrix can be detected in both conditions, consistent with results from

Liu et al. [?]. The volume fractions of carbides, Laves phase, and δ-phase are small [?], making them difficult to detect by XRD. Additionally, the γ phase is coherently precipitated with the γ matrix [?], so γ diffraction peaks are masked by γ-phase peaks.

2.2 Mechanical Properties

presents the room-temperature tensile properties of LSF Inconel 718 alloy at different heat treatment stages. After the three-stage heat treatment, the alloy's tensile strength and ductility both satisfy wrought standards.

[Figure 4: see original paper] shows the tensile fracture morphology of heat-treated LSF Inconel 718 alloy specimens. The fracture surface consists of two main regions: a fibrous zone and a shear lip zone. The fibrous zone is located primarily at the center of the fracture, containing many fibrous peaks with slopes at approximately 45° to the tensile axis. The shear lip zone at the fracture edges is relatively flat, occupies a smaller area, and is oriented at approximately 45° to the principal stress direction. Numerous fine secondary cracks are observed on the fracture surface, mostly distributed in the fibrous zone with lengths ranging from 200–500 m. As shown in Fig. 4b, the fibrous zone of the heat-treated LSF Inconel 718 alloy specimen contains many dimples approximately 1–2 m in size. In some regions, several small dimples connect to form larger dimples, consistent with observations by Lin et al. [?] in nickel-based superalloy tensile fractures, indicating good ductility of the LSF Inconel 718 alloy. Large dimples typically reach 6–8 m and mainly form through microvoid coalescence initiated at γ strengthening phase particles. Fine particles can also be observed within some dimples [?], resulting from second-phase particle fracture under shear stress and subsequent dimple formation during deformation. Fan-shaped tear ridges are visible on dimple walls, indicating that cracks originated at the fan handle and propagated along tear ridge directions before interacting with adjacent dimples under shear stress to form new dimple ridges. Several parallel fine serpentine slip patterns are also observed in Fig. 4b. When the matrix has good ductility, mutual constraint between grains of different orientations in Inconel 718 alloy causes dislocations to glide along several intersecting slip planes, creating curved stripes. Dimples formed after slip separation exhibit serpentine morphology [?]. The presence of serpentine slip patterns indicates that more slip systems can be activated when stress concentrations form, demonstrating excellent ductility of the LSF Inconel 718 alloy. Additionally, some elongated oval dimples are observed on tear ridges of large dimples. These form during tensile fracture of Inconel 718 alloy when large dimples develop first and their free surfaces connect with small dimples during growth, causing the small dimples to attach completely to the large dimples. When the large dimple tears, the small dimples undergo further plastic deformation, enhancing the alloy's fracture resistance.

2.3 Dislocation Configuration

After heat treatment, LSF Inconel 718 alloy exhibits fine δ-phase precipitated along grain boundaries and dispersive γ phase within grains. For such precipitation-strengthened superalloys, mechanical properties are closely related to second phases, and interactions between second phases and dislocations reflect the strengthening mechanisms. Therefore, investigating dislocation configurations after tensile fracture is essential for clarifying the strengthening mechanisms of heat-treated LSF Inconel 718 alloy.

2.3.1 γ Phase [Figure 5: see original paper] shows bright-field and dark-field TEM images and the corresponding diffraction pattern of γ strengthening phase in heat-treated LSF Inconel 718 alloy. As seen in Fig. 5a, γ phase dispersively distributes in the γ matrix as circular discs. Lattice misfit between γ phase and γ matrix generates stress fields that hinder dislocation motion. In conventionally processed Inconel 718 alloy, the primary mechanism for γ phase blocking dislocation motion is dislocation shearing through γ phase [**?**], which also occurs in heat-treated LSF Inconel 718 alloy. When γ phase precipitation is relatively sparse (region A in Fig. 5a), dislocation lines are relatively straight, and the interaction mechanism is primarily shearing. Fig. 5b shows that after interaction with γ phase, some dislocations cut through γ phase while others only partially shear it, stopping within the γ phase. Fig. 5c shows the [111] zone axis electron diffraction pattern of γ phase. The crystal orientation determined from diffraction spots indicates that the slip direction of dislocations shearing γ phase is primarily $\langle 11\bar{0} \rangle$, consistent with observations by Sundararaman et al. [**?**]. When dislocations shear γ phase, some zigzag segments appear between two dislocation lines with the same orientation, possibly caused by cross-slip of dislocations on γ phase. Dislocation shearing through γ phase increases energy, thereby providing strengthening. Shearing creates new interfaces, increasing interfacial energy. After dislocation passage, anti-phase boundary energy is generated, and the difference in shear modulus between γ phase and matrix also causes energy increase. As seen in Fig. 5a, when γ phase precipitation is dense (region B in Fig. 5a), dislocations cannot easily shear through, and the interaction mechanism transitions to the Orowan bypass mechanism. When γ phase coarsens, dislocation lines bend around γ phase under applied stress. This bending increases lattice distortion energy in the dislocation-affected zone, creating greater resistance to dislocation motion and increasing slip resistance. Furthermore, regions with higher γ phase precipitation density also exhibit higher dislocation density because bypassing requires more energy than shearing. Dislocations experience greater resistance in regions with dense γ phase precipitation, making dislocation pile-up more likely. In high dislocation density regions, dislocations become entangled, further impeding dislocation motion and contributing to increased strength of Inconel 718 alloy.

2.3.2 δ Phase [Figure 6: see original paper] shows a bright-field TEM image and corresponding $[\bar{1}03]$ zone axis electron diffraction pattern of δ-phase precipitated in heat-treated LSF Inconel 718 alloy. Two different slip bands are observed at the δ-phase: at edge A of the δ-phase, dislocation slip direction is [341], while within the δ-phase at B, slip direction is [$\bar{3}4\bar{1}$].

In conventionally wrought Inconel 718 alloy, δ-phase typically appears as slender needles approximately 100 nm wide, making it easily sheared by dislocations during deformation. However, in heat-treated LSF Inconel 718 alloy, δ-phase is short rod-shaped with width of about 1 m, making dislocation pile-up more likely and thus more effective at blocking dislocation motion. In the heat-treated alloy, δ-phase edges are sheared by dislocations, creating slip steps (region A in Fig. 6a) that increase interfacial area and energy. This suggests that dislocation shearing through δ-phase may cause atomic misalignment, increasing misalignment energy. In region A of Fig. 6a, the distance between adjacent dislocation lines gradually increases as they shear δ-phase along [341] direction, caused by repulsion between dislocations with the same Burgers vector. When dislocations pile up at δ-phase, the leading dislocation arrives first, while subsequent dislocations become increasingly spaced due to repulsive forces [**?**]. Additionally, dislocations with slip direction [$\bar{3}4\bar{1}$] interact with those having slip direction [341], producing jogs and kinks. Within the δ-phase (region B in Fig. 6a), dislocations shear δ-phase but do not cut through it completely, stopping within the δ-phase. This indicates that the larger δ-phase in the heat-treated condition provides significant resistance to dislocation motion.

2.3.3 Carbides Besides γ and δ phases, carbides are also common in heat-treated LSF Inconel 718 alloy. Liu et al. [**?**] reported that dislocations can tangle around carbides, hindering dislocation motion and increasing shear resistance to strengthening phases, thereby improving alloy strength. He et al. [**?**] also noted strong interactions between carbides and dislocations that impede dislocation motion through pinning and drag effects. [Figure 7: see original paper] shows a bright-field TEM image and EDS analysis of carbides in heat-treated LSF Inconel 718 alloy. The phase has relatively straight boundaries and appears blocky. EDS analysis (Fig. 7b) shows high Ti and Nb content, identifying it as (Ti, Nb)C carbide. Morphologically, traces of dislocation shearing are visible within the carbide, and slip bands indicate dislocation pile-up inside the carbide.

Among these strengthening phases, γ phase dominates strengthening of LSF Inconel 718 alloy, primarily because its volume fraction can reach 50–65% with a body-centered tetragonal structure, large lattice parameters, and strong coherent strain strengthening [**?**]. In contrast, the volume fractions of δ-phase and carbides are much lower than that of γ phase.

Conclusions

1. After high-temperature solution treatment, δ-phase aging, and double aging, the microstructure of LSF Inconel 718 alloy transforms from initial columnar grains to equiaxed grains with grain refinement. Laves phase is

completely dissolved, needle-like δ-phase precipitates along grain boundaries, and γ strengthening phase dispersively precipitates in the matrix.

2. After heat treatment, the alloy exhibits tensile strength of 1351 MPa, yield strength of 1184 MPa, elongation of 17.20%, and reduction of area of 23.70%, with strength and ductility comparable to wrought standards.
3. The tensile fracture surface of the heat-treated alloy consists only of fibrous and shear lip zones. The fibrous zone contains many dimples nucleated at γ phase, with serpentine and fan patterns observed within some dimples. Additionally, small oval dimples are present on tear ridges of large dimples.
4. The strengthening mechanisms of the heat-treated alloy depend primarily on second phases: γ phase, δ-phase, and carbides. γ phase precipitation is denser in heat-treated LSF Inconel 718 alloy than in conventional wrought material. In regions with lower γ phase density, dislocations typically shear through γ phase, while in regions with higher density, γ phase is more prone to coarsening, and the strengthening mechanism transitions from shearing to Orowan bypassing. Bypassing requires more energy, so dislocation motion encounters greater resistance in dense γ phase regions, making pile-up more likely and resulting in higher dislocation density. The δ-phase in heat-treated LSF Inconel 718 alloy is larger than in wrought material, making it more difficult to shear. Dislocation pile-up at δ-phase is hindered by both dislocation repulsion and δ-phase resistance, making dislocation motion near δ-phase more difficult than in wrought material. Strong interactions between carbides and dislocations impede dislocation motion through pinning and drag effects.

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