Effect of Homogenization Degree of Superalloy Ingot on Recrystallization During Breakdown Hot Deformation (Postprint)

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Abstract

Utilizing optical microscopy (OM), scanning electron microscopy (SEM), and crack propagation rate testing, the microstructural characteristics under various homogenization degrees and the hot deformation behavior of these microstructural states in GH4740H, GH4738, GH3625, and 690 alloys were analyzed, and the correlation between alloy homogenization process parameters and recrystallization was systematically investigated. The results demonstrate that alloy homogenization must balance the improvement in hot ductility arising from dendritic segregation elimination against the plasticity degradation during subsequent hot deformation caused by grain growth and intensified oxidation; the residual interdendritic regions remaining from incomplete homogenization provide recrystallization nucleation sites, thereby enhancing the recrystallization nucleation rate. Under identical hot deformation conditions, the recrystallization extent in specimens with residual dendritic structures is significantly higher than that in fully homogenized specimens without dendritic structures. Therefore, the proposal of a superalloy homogenization breakdown control methodology based on partial homogenization regimes is justified.

Full Text

Effect of Homogenization Extent on Recrystallization During Hot Deformation of Superalloy Ingots in Cogging Process

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Abstract

The microstructural characteristics and hot deformation behaviors of GH4740H, GH4738, GH3625, and 690 alloys under different homogenization conditions were investigated using optical microscopy (OM), scanning electron microscopy (SEM), and crack propagation rate testing. The correlation between homogenization parameters and recrystallization was systematically studied. The results indicate that homogenization must balance the improvement in hot ductility from eliminating dendritic segregation against the detrimental effects of grain coarsening and oxidation on subsequent hot workability. Residual interdendritic regions remaining after incomplete homogenization provide nucleation sites for recrystallization, thereby increasing the nucleation rate. Under identical hot deformation conditions, specimens retaining dendritic structures exhibit significantly higher recrystallization fractions than fully homogenized specimens without dendritic structures. Based on these findings, a homogenization-cogging control method employing partial homogenization is proposed for superalloys.

Keywords superalloy, homogenization, cogging, dendrite

Introduction

Wrought superalloys, particularly those that are difficult to deform, exhibit severe compositional segregation and segregation phases after solidification due to their high alloying content, resulting in poor hot workability [1~3]. Consequently, high-temperature diffusion annealing (homogenization heat treatment) at specific temperatures and durations is required to eliminate elemental dendritic segregation before ingot breakdown. Hot deformation then fractures the coarse columnar grains to obtain uniform, controllable recrystallized microstructures, providing high-quality billets for subsequent forging operations [4~8]. As a critical link between casting and forging, the homogenization and cogging processes play a pivotal role in the overall production chain of wrought superalloys.

Currently, domestic research on superalloy component manufacturing has focused extensively on downstream hot forging and heat treatment operations [9~15], accumulating considerable technical expertise. Upstream smelting has also advanced significantly through the introduction of advanced equipment and technologies [16,17]. Although previous studies have guided homogenization process design by establishing diffusion equations to simulate elemental segregation [1,2,7,18] and developed hot deformation microstructure models for cogging simulation [4,19~21], practical challenges persist: homogenization processes are time-consuming and energy-intensive (typically conducted at approximately 1200 °C for 20–70 h), cogging is prone to cracking, and the resulting grain structure is often coarse, leading to low final product yield.

In the fabrication of critical hot-section components such as turbine disks, the controllable degree of grain refinement through recrystallization during forging is limited. Considering microstructural heredity throughout the thermomechanical processing chain, the initial grain structure at the upstream stage significantly influences subsequent grain evolution. The grain size produced during homogenization thus becomes the origin of grain size control, while the cogging process represents the first stage of grain refinement through recrystallization. This raises the question of whether homogenization can be controlled to prevent excessive grain growth while simultaneously improving hot ductility and recrystallization fraction for effective grain refinement. Therefore, systematic investigation of the relationship between homogenization heat treatment and hot deformation behavior is essential.

Microstructural changes during homogenization include: (1) elimination of dendritic structures through uniform diffusion of segregated elements; (2) dissolution of segregation-induced precipitates; (3) grain growth; and (4) surface oxidation. The impact of these homogenization-induced microstructural changes on subsequent hot deformation ductility constitutes the core consideration in linking homogenization with hot deformation. This study examines the microstructural characteristics of alloys under various homogenization conditions and their corresponding hot deformation behaviors to explore how homogenization-induced changes affect subsequent hot deformation, providing experimental and theoretical foundations for process improvement.

Experimental Materials and Methods

The experimental materials included GH4740H, GH4738, GH3625, and 690 alloys. GH4740H and 690 alloys were produced as 350 mm and 510 mm diameter ingots, respectively, via vacuum induction melting plus electroslag remelting. GH3625 alloy was obtained as a 350 mm diameter ingot through vacuum induction melting, while GH4738 alloy was produced as a 170 mm diameter ingot via vacuum induction plus vacuum arc remelting. The chemical compositions of the four alloys are listed in . All test specimens were sectioned from the R/2 position (where R is the ingot radius) of the transverse cross-section.

For GH4740H alloy, homogenization was performed at 1110 °C and 1170 °C for 8, 24, and 48 h. Samples were mounted and carbon-coated for examination using a TSM-6510 SEM with energy-dispersive spectroscopy (EDS) to characterize oxidation cross-section morphology, oxide phase composition, and oxide layer thickness.

GH4738 alloy was homogenized at 1150 °C for 3, 12, 24, and 50 h. To investigate the effect of homogenization extent on recrystallization, Gleeble hot compression tests were conducted at 1150 °C with a strain rate of 0.1 s^{-1} and 30% deformation on specimens subjected to different homogenization durations.

Hot deformation behavior of as-cast GH3625 specimens was studied via Gleeble compression testing. Samples were heated to 1250 °C at 15 °C/s, held for 5 min, cooled to 1100 °C at 15 °C/s, and compressed to 50% strain at 0.01 s^{-1} before water quenching. To compare the effects of hot deformation and holding on segregation and recrystallization, three conditions were examined: as-cast, soaked (1250 °C for 5 min, water quenched), and deformed (water quenched after final deformation).

For 690 alloy, homogenization temperatures ranged from 1150 °C to 1240 °C with holding times from 8 to 60 h. Crack propagation rate tests were performed on specimens homogenized at 1150 °C/60 h, 1180 °C/30 h, 1220 °C/8 h, and 1240 °C/30 h. Standard compact tension specimens were prepared according to JB/T8189-1999. Loading employed a trapezoidal waveform with 15 s loading/unloading times and 90 s hold time, maintaining load error below 1%.

Microstructures were examined using a 9XB-PC optical microscope (OM), JSM-6510 SEM, and SUPRA55 field-emission SEM (FSEM). EDS multi-point measurements across dendritic and interdendritic regions determined elemental segregation indices. Metallographic specimens were prepared by mechanical grinding and polishing, followed by etching in a solution of 2.5 g $KMnO_4$ + 10 mL H_2SO_4 + 90 mL H_2O at boiling for 20 min. SEM specimens were electrolytically etched using aqua regia-glycerol (glycerol:HCl:HNO_3 = 5:3:1 by volume) at 5 V for 10 s. FSEM specimens were prepared by electrolytic polishing (20% H_2SO_4 + 80% CH_3OH) followed by electrolytic etching (2.5 g CrO_3 + 10 mL H_2SO_4 + 90 mL H_3PO_4).

2.1 Effect of Homogenization Extent on Microstructure and Properties

[Figure 1: see original paper] shows the dendritic segregation structure at the R/2 position of a 510 mm diameter as-cast 690 alloy ingot. EDS analysis revealed that Ti and Cr were positive segregation elements (enriched in interdendritic regions), while Fe was a negative segregation element (enriched in dendrite cores). Statistical measurements yielded average segregation indices (interdendritic concentration/dendrite core concentration) of 3.392 for Ti, 1.097 for Cr, and 0.917 for Fe, indicating Ti as the primary segregating element.

[Figure 2: see original paper] illustrates the residual segregation index and grain size evolution in 690 alloy under various homogenization conditions. At constant temperature, increasing holding time gradually reduces dendritic segregation. At constant time, elevated homogenization temperature also facilitates segregation elimination. After 60 h at 1150 °C, residual dendritic segregation remained, whereas at 1180 °C for 30 h, only minor segregation persisted. While higher temperatures and longer durations effectively reduce residual segregation index, process design must not solely pursue complete segregation elimination. Homogenization treatments achieving a residual segregation index near 0.2 (the engineering threshold for acceptable segregation elimination) include 1180 °C/30-60 h, 1220 °C/30-40 h, and 1240 °C/30 h. However, these conditions produce markedly different grain sizes.

Crack propagation resistance correlates with multiple mechanical properties including strength and toughness [22]. Therefore, measuring crack growth rates in specimens subjected to different homogenization treatments provides another metric for evaluating homogenization effectiveness. [Figure 3: see original paper] and [Figure 4: see original paper] present crack growth rate (da/dN) versus stress intensity factor range (ΔK) curves and fracture morphologies for 690 alloy under various homogenization conditions. The 1150 °C/60 h specimen exhibited the fastest crack growth rate with interdendritic fracture features, confirming residual segregation. The 1180 °C/30 h specimen showed the lowest crack growth rate, with extensive fatigue striations and dimples indicating significantly improved properties. However, further temperature increase to 1240 °C/30 h accelerated crack growth, demonstrating that while higher temperatures promote segregation elimination, excessive grain coarsening facilitates intergranular crack propagation. The 1220 °C/8 h specimen also exhibited rapid crack growth, indicating insufficient homogenization time despite the temperature increase.

Research on G3 alloy [23] demonstrated that grain size increases rapidly with heating temperature, initially improving hot ductility but causing a sharp ductility drop when grains become excessively large. Additionally, the post-homogenization grain size serves as the initial condition for subsequent thermomechanical processing, directly influencing process parameter selection and final microstructure. Studies on GH2132 alloy [24] showed that achieving the same final grain size required approximately 20% deformation for material with initial grain size grade 1, but only 8% for grade 3. Larger initial grain size necessitates greater deformation, increasing cracking risk. When deformation is insufficient, microstructural heredity is pronounced because dynamic recrystallization occurs primarily through grain boundary migration, where initial grain size effects are significant. Thus, substantial deformation is required to eliminate the hereditary influence of coarse initial grains.

In summary, while dendritic segregation elimination during homogenization improves hot workability, concurrent grain growth adversely affects ductility and complicates grain refinement through hereditary effects. Therefore, homogenization process design must consider both segregation elimination benefits and negative impacts from grain coarsening.

2.2 Effect of Homogenization Extent on High-Temperature Oxidation

Besides elemental segregation and grain growth, high-temperature oxidation during prolonged homogenization must also be considered. Due to the high temperatures and extended durations involved, superalloy ingots undergo oxidation. The oxide layer typically consists of inner and outer layers: a continuous external layer enveloping the alloy surface and a dispersed internal layer.

[Figure 5: see original paper] shows the dendritic segregation structure at the R/2 position of a 350 mm diameter as-cast GH4740H ingot. EDS analysis identified Al, Ti, Fe, and Nb as positive segregation elements, while Cr and Co were negative segregation elements. Ti and Nb exhibited the most severe segregation with indices of 4.44 and 3.02, respectively.

After 8 h at 1110 °C, a non-dense, incomplete external Cr oxide layer formed on the surface, with internal Al and Ti oxides distributed within the matrix [Figure 6a: see original paper]. After 24 h, a thick, dense external Cr oxide layer developed, though poorly bonded to the substrate. Internal Al and Ti oxides predominantly existed along grain boundaries. Localized spalling of the external layer led to more severe internal oxidation. [Figure 7: see original paper] shows SEM images and elemental maps after 48 h at 1110 °C, clearly revealing Al and Ti oxides on grain boundaries, indicating extensive internal oxidation penetrating inward from the spalled surface. During homogenization, when the Cr oxide layer overgrows under certain conditions (temperature/time), severe Cr depletion at the oxide-metal interface weakens bonding, facilitating spalling and accelerating internal oxidation.

At 1170 °C, cross-sectional morphologies after 8 and 48 h are shown in [Figure 8: see original paper]. Even after short-term (8 h) oxidation, a continuous, dense external oxide film formed. Internal oxide particles were present, with some extending as elongated features into the substrate. EDS analysis indicated the external layer was Cr-rich with minor Ti (Cr-Ti complex oxide), while the dominant black internal oxides consisted primarily of Al with negligible Ti. Unlike the dispersed irregular particles at 1110 °C, internal oxides at 1170 °C exhibited aligned, elongated morphologies. Comparison of [Figure 8a: see original paper] and [8b] shows that without external layer spalling, the elongated internal oxides extended significantly deeper with increasing time, substantially expanding the affected zone.

[Figure 9: see original paper] shows the oxidation layer thickness evolution for GH4740H at 1170 °C, including both external oxide and affected zone thicknesses. Despite thickness reduction due to spalling in some specimens, the overall trend shows significant thickness increase with time. High-temperature oxidation during homogenization degrades near-surface mechanical properties, impairing hot workability. Surface machining (turning) is required before cogging, with the removal depth depending on internal oxidation severity. Therefore, oxidation behavior during high-temperature, long-duration heat treatment must be considered in homogenization process design.

2.3 Microstructural Evolution During Hot Deformation

[Figure 10: see original paper] compares microstructures of GH3625 alloy in as-cast, soaked, and deformed conditions. The as-cast structure contained dense dendritic segregation [Figure 10a: see original paper]. EDS analysis at the R/2 position identified Cr, Mo, and Nb as primary segregating elements with indices of 0.85, 1.14, and 3.59, respectively, with Nb showing the highest segregation. After soaking, minor dendrite dissolution occurred [Figure 10b: see original paper], indicating that heating promotes localized elemental diffusion. Following hot compression, dendritic structures were significantly degraded compared to the soaked condition, with interdendritic spacing reduced along the compression direction and dendrites themselves compressed [Figure 10c: see original paper].

Notably, interdendritic regions served as important recrystallization nucleation sites. During compression, strain incompatibility between adjacent dendrites accumulated high distortion energy in interdendritic regions, promoting preferential recrystallization nucleation.

Statistical analysis of segregation indices for Cr, Mo, and Nb in different conditions [Figure 11: see original paper] shows that 5 min at high temperature reduced Nb segregation significantly, with Mo also decreasing and Cr becoming nearly uniform. Hot deformation further reduced Nb segregation substantially. The deformed microstructure [Figure 12: see original paper] confirms these results through EDS mapping, demonstrating that hot deformation alleviates segregation while interdendritic regions remain preferential sites for recrystallization.

2.4 Effect of Homogenization Extent on Recrystallization Behavior

Since as-cast microstructures exhibit poor hot ductility, homogenization is mandatory before practical cogging. Therefore, investigating hot deformation behavior under various homogenization conditions is essential.

The as-cast microstructure of GH4738 alloy [Figure 13a: see original paper] showed segregation indices of 1.17 for Al, 2.19 for Ti, and 1.04 for Mo (positive segregation), and 0.85 for Cr and 0.87 for Co (negative segregation), with Ti being the most segregated element.

Microstructural evolution after homogenization at 1150 °C for 12, 24, and 50 h is shown in [FIGURE:13b-d]. After 12 h, dendritic structure was significantly degraded (Ti segregation index reduced to 1.39). After 24 h, dendrites were essentially eliminated. Grain growth was modest after 12 h but became pronounced after 24 h, with further coarsening after 50 h.

True stress-true strain curves for the four homogenization conditions under compression at 1150 °C, 0.1 s^{-1}, 30% strain [Figure 14: see original paper] show that as-cast material had the highest flow stress. Flow stress decreased with increasing homogenization but increased after over-homogenization. The as-cast curve exhibited softening after a peak strain of 0.12. Similarly, the 12 h homogenized specimen showed pronounced softening after peaking at 0.15 strain, with flow stress dropping below that of the 50 h specimen beyond 0.25 strain and approaching the 24 h specimen. The 24 h and 50 h specimens, with essentially eliminated dendrites, showed relatively stable flow curves.

Metallographic examination of highly deformed central regions [Figure 15: see original paper] revealed recrystallization in all conditions, with recrystallization fraction decreasing in the order: 12 h homogenized > as-cast > 24 h homogenized > 50 h homogenized. This trend correlates with the flow stress behavior, demonstrating that incompletely homogenized microstructures with residual dendrites undergo more extensive recrystallization and softening than fully or over-homogenized structures.

[Figure 16: see original paper] clearly shows recrystallization nucleation preferentially occurring in interdendritic regions. In low-strain zones, initial recrystallization appears exclusively in interdendritic areas, while in high-strain zones, most recrystallized grains reside in interdendritic regions, with minor contributions from isolated carbide particles.

FESEM observations [Figure 17: see original paper] reveal that segregation differences persist after deformation. In as-cast material, severe Ti segregation led to extensive γ' precipitation in interdendritic regions, with local γ-γ' eutectic phases. After 12 h homogenization, γ' precipitation was confined to interdendritic centers, with improved regions near dendrite boundaries showing no γ' precipitation. The as-cast dense dendritic structure results in bulk deformation of dendrite-interdendrite assemblies, whereas after 12 h homogenization, enlarged interdendritic regions accommodate more deformation, promoting recrystallization nucleation in high-distortion-energy interdendritic zones. Consequently, partially homogenized (12 h) material shows higher recrystallization fractions and more pronounced softening than as-cast material. Over-homogenized material lacks dendrites and possesses coarse grains, eliminating interdendritic deformation accommodation.

EDS quantitative analysis of Ti segregation after deformation shows reduced segregation in all conditions compared to as-cast, with central highly-deformed regions showing lower segregation indices than edge dead zones, confirming that hot deformation improves segregation.

Hot deformation also alters dendrite morphology, reducing primary and secondary dendrite arm spacings. [Figure 18: see original paper] shows that compression significantly "flattened" dendrites along the compression direction, markedly reducing spacings.

The elemental distribution during homogenization follows Equation (1) [25]:

$$C(x) = \bar{C} + \Delta C_0 \cos\left(\frac{\pi x}{L}\right) \exp\left(-\frac{\pi^2 D t}{L^2}\right)$$

where $C(x)$ is the element mass fraction at position x, $\bar{C}$ is the average mass fraction, ΔC_0 is the difference between maximum/minimum and average concentrations, L is dendrite spacing, D is the diffusion coefficient, and t is homogenization time. Considering only maximum ($C_{\max}$) and minimum ($C_{\min}$) concentrations after homogenization, the residual segregation index d is defined as:

$$d = \frac{C_{\max} - C_{\min}}{C_0^{\max} - C_0^{\min}} = \exp\left(-\frac{\pi^2 D t}{L^2}\right)$$

where $C_0^{\max}$ and $C_0^{\min}$ are the original as-cast concentrations. Assuming original secondary dendrite arm spacing L_1 and compressed spacing $L_2 = L_1/2$, if both structures are homogenized at the same temperature until the deformed specimen’ s composition decays to $1/e$:

$$\exp\left(-\frac{\pi^2 Dt}{L_2^2}\right) = \exp\left(-\frac{4\pi^2 Dt}{L_1^2}\right) = \frac{1}{e}$$

The composition decay in the undeformed as-cast specimen during the same time would be:

$$\exp\left(-\frac{\pi^2 Dt}{L_1^2}\right) = \exp\left(-\frac{1}{4}\right) \approx 0.779$$

Thus, when the deformed specimen’ s composition has decayed by $(1-1/e) \times 100\% = 63.2\%$, the as-cast specimen has only decayed by $(1-0.779) \times 100\% = 22.1\%$. This demonstrates that hot deformation significantly reduces diffusion distances, facilitating subsequent segregation elimination.

Conclusions

Comprehensive analysis reveals distinct differences between fully/partially homogenized microstructures and their recrystallization behavior:

1. **Fully/over-homogenized structures** eliminate microsegregation and dendrites but exhibit pronounced grain growth and severe oxidation, causing ductility to first increase then decrease. **Partially homogenized structures** improve segregation while retaining residual dendrites, with modest grain growth and less oxidation.

2. Hot deformation improves segregation, modifies dendrite morphology, reduces dendrite spacings, and significantly shortens diffusion distances, thereby accelerating subsequent segregation elimination.

3. **Incomplete homogenization** retaining residual dendrites promotes interdendritic deformation accommodation and recrystallization nucleation. Under identical deformation conditions, specimens with residual dendrites exhibit significantly higher recrystallization fractions than fully homogenized, dendrite-free specimens.

4. Considering economic factors, microstructural control, and cracking sensitivity, a homogenization-cogging approach based on partial homogenization is reasonable and feasible. Further work should comprehensively evaluate plasticity evolution, deformation resistance, and cracking sensitivity to optimize the process economically.

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Note: Figure translations are in progress. See original paper for figures.