Analysis of Recrystallization Mechanisms During Hot Working of Nickel-Based Alloys for Ultra-Supercritical Power Plants - Postprint

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Abstract

This study investigates a novel nickel-based alloy for ultra-supercritical power plants. Combining the cold working and hot working of alloy tubes with a series of hot compression deformation and solution annealing experiments, and using optical microscopy and transmission electron microscopy, the dynamic and static recrystallization behaviors of the alloy during hot processing were systematically analyzed. The results show that the dynamic recrystallization process of the novel nickel-based alloy is dominated by discontinuous dynamic recrystallization nucleated through grain boundary bowing, while static recrystallization is primarily governed by a strain-induced grain boundary migration nucleation mechanism. Furthermore, the stepped grain boundaries with different morphologies generated during dynamic and static recrystallization processes essentially serve to deviate the surface from low-index planes, ensuring that more grain boundary surfaces are low-energy close-packed planes to reduce grain boundary energy; their morphology depends on the crystallographic relationship of the grain boundary plane and the Burgers vector of grain boundary dislocations; and their presence can also promote grain boundary migration, thereby accelerating the recrystallization process. Moreover, after the recrystallization process is completed, stepped grain boundaries are partially retained to reduce interfacial energy and continue promoting subsequent grain growth.

Full Text

Analysis of Recrystallization Mechanisms in Hot Working Processes of a Nickel-Based Alloy for Ultra-Supercritical Power Plant Application

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Abstract

A nickel-based alloy for ultra-supercritical power plant tubing was investigated. Combining the cold/hot working processes of the tubes with a series of compression hot deformation and solution annealing experiments, the dynamic and static recrystallization behaviors during hot working were analyzed using OM and TEM. The results showed that dynamic recrystallization was dominated by discontinuous dynamic recrystallization nucleated via grain boundary bulging, while static recrystallization was primarily governed by strain-induced grain boundary migration. Furthermore, step grain boundaries with different morphologies formed during dynamic and static recrystallization serve to deviate the surface from low-index planes, ensuring more grain boundary interfaces are low-energy close-packed planes, thereby reducing grain boundary energy. Their morphology depends on the crystallographic relationship of the grain boundary plane and the Burgers vector of grain boundary dislocations. The presence of step grain boundaries can also promote grain boundary migration and accelerate recrystallization. Moreover, after complete recrystallization, step grain boundaries remain partially to reduce interfacial energy and continue promoting subsequent grain growth.

Keywords: nickel-based alloy, recrystallization, step grain boundary

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Introduction

phase precipitation-strengthened nickel-based wrought superalloys exhibit high temperature strength, good oxidation resistance, and hot corrosion resistance, and have been widely used in critical high-temperature components such as aero-engine turbine disks [1]. These excellent properties are determined by the alloy microstructure and are closely related to the recovery and recrystallization processes of metals [2~5]. To date, numerous studies have investigated the recrystallization behavior of nickel-based alloys. Singh et al. [6] studied the recrystallization behavior of cold-rolled IN718 alloy with 30% and 50% reduction after annealing at 1130-1214 K for various times. Wang et al. [7] investigated the dynamic recrystallization nucleation mechanisms and the effect of twins on dynamic recrystallization in GH4586 alloy at deformation temperatures of 1273 and 1373 K with strain ranging from 20% to 60%. Liu et al. [8,9] used a cellular automaton model to study prediction methods for dynamic and static recrystallization behaviors in a nickel-based alloy. However, crystal growth mechanisms often involve the movement of grain boundaries and defects, accompanied by dislocation and/or step characteristics [10~12]. Most existing research has focused on the pinning effect of carbides on grain boundaries during migration, or on serrated grain boundaries formed to alleviate discontinuous segregation of elements at grain boundaries and the resulting unbalanced strain, as well as the influence of serrated grain boundaries on grain boundary motion and mechanical properties [13~19]. Studies on the formation mechanisms of straight step grain boundaries without phase segregation are relatively scarce.

This work investigates a nickel-based alloy for 700 ℃ advanced ultra-supercritical power plant tubing, focusing on its recrystallization behavior during hot working processes. The formation mechanism of step grain boundaries during dynamic and static recrystallization is analyzed from a materials science perspective, providing a theoretical basis for microstructure control during hot working of corrosion-resistant nickel-based alloys.

Experimental

The experimental material was a backup material for 700 ℃ advanced ultra-supercritical power plants developed by the University of Science and Technology Beijing [20,21], with chemical composition (mass fraction, %) of: C 0.02-0.06, Cr 20.0-23.0, Co 12.0-14.5, Mo 4.0-6.0, W 0.5-1.5, Nb 1.3-1.6, Ti 1.3-1.6, Al 1.3-1.6, Ni balance. The alloy was melted using vacuum induction melting (VIM) + electroslag remelting (ESR) double melting process, then subjected to high-temperature homogenization treatment and formed into a 120 mm diameter bar using a 3500T press. The bar was then extruded into a hollow tube on a 3600T extrusion machine, and finally cold-rolled into a tube with outer diameter of 44.5 mm and thickness of 10 mm through multiple passes.

To investigate the dynamic recrystallization mechanism of this alloy, cylindrical specimens with diameter of 10 mm and length of 15 mm were cut axially from the forged bar. Compression tests were conducted on a Gleeble-1500 thermal simulator, with the process schematic shown in [Figure 1: see original paper]. Deformation temperatures were 1000, 1050, 1100, 1150, and 1200 ℃, strain rates were 0.01, 1.0, 10, and 20 s^{1}, and deformation amount was 50%. After hot deformation, specimens were sectioned along the compression axis. The cut surfaces were mechanically ground and polished, then boiled in a solution of 2.5 g KMnO + 10 mL H SO + 90 mL H O for 5 min. After cleaning and drying, the grain structures were observed using a DMR optical microscope (OM). From the other half of the longitudinal section, 0.5 mm thick slices were cut, mechanically thinned to 50 m, punched into 3 mm diameter discs, twin-jet electropolished in 5% perchloric acid-ethanol solution (volume fraction), and then examined using a Tecnai G2 F30 S-TWIN transmission electron microscope (TEM).

Block specimens of 10 mm × 10 mm × 5 mm were cut radially from the cold-rolled tube (with the 10 mm × 10 mm surface perpendicular to the radial direction) for solution annealing. Solution temperatures were selected as 1100, 1120, 1130, 1140, 1150, 1160, 1170, and 1180 ℃, holding times were 5, 10, 20, 40, and 60 min, and cooling was performed by water quenching. The 10 mm × 10 mm surfaces of the cold-rolled and various solution-treated specimens were prepared using the same method described above, and OM and TEM were used to observe the static recrystallization behavior.

2.1 Microstructural Characteristics and Dynamic Recrystallization Mechanism After Hot Deformation

[Figure 2: see original paper] shows the longitudinal section microstructure of the forged bar. The original microstructure of the forged bar was quite inhomogeneous, containing some unrecrystallized elongated grains. [Figure 3: see original paper] presents the recrystallized microstructures of specimens deformed to 50% under different temperatures and strain rates. After compression deformation under various conditions, the alloy underwent significant recrystallization, and the original forged microstructure was essentially replaced by uniform, fine dynamic recrystallization structures. Additionally, lower strain rates resulted in more complete recrystallization, with strain rate having minimal effect on grain size, while grain size increased significantly with deformation temperature. Dynamic recrystallization grains at a strain rate of 0.01 s ^{1} exhibited obvious grain boundary bulging.

[Figure 4: see original paper] shows TEM images of the alloy under different hot deformation conditions. As shown in Figs. 4a-c, grain boundary bulging was observed under various compression conditions, indicating that the dynamic recrystallization mechanism was primarily discontinuous dynamic recrystallization. In Fig. 4b, the bulging portion was intercepted by twins, which can promote recrystallization [7,22,23]. Additionally, at high temperature and high strain rate, features of continuous dynamic recrystallization through subgrain coalescence and growth were also present, as shown in Fig. 4d.

Nickel-based alloys are low stacking fault energy materials. During hot deformation, dislocation cross-slip and climb are difficult, recovery processes are hindered, and dynamic recrystallization dominates [11]. Deformation of polycrystals is inhomogeneous; due to differences in grain orientation, grains with different orientations experience varying strain levels. Grains with higher strain have higher dislocation density, while those with lower strain have lower dislocation density, creating a significant dislocation density difference across grain boundaries during deformation. This causes original grain boundaries to bulge from the low dislocation density side toward the high density side, forming a stable interface after reaching a certain size. The protruding tongues in Figs. 4a-c nucleated at grain boundaries where dislocation densities differed on either side. According to classical strain-induced grain boundary migration theory [3], a viable nucleus can only form when the following relationship is satisfied:

where ΔE is the difference in stored energy per unit volume across the bulged grain boundary, _b is the grain boundary energy, and L is half the length of the original grain boundary corresponding to the bulged portion.

At the same strain level, lower strain rates involve longer deformation times, allowing more complete progression of discontinuous dynamic recrystallization dominated by grain boundary bulging. Recrystallized grains also have sufficient time to grow and can undergo grain boundary bulging again, producing a new round of discontinuous dynamic recrystallization. Therefore, OM and

TEM analyses reveal that the primary recrystallization nucleation mechanism at low strain rates is discontinuous dynamic recrystallization dominated by grain boundary bulging. As strain rate increases, shorter deformation times prevent dislocation annihilation in many regions, and the limited time for dynamic recrystallization inhibits grain growth, leaving scattered dislocations within grains, as shown in Figs. 4c and d.

Under the same strain rate conditions, deformation at the lower temperature of 1050 ℃ results in weaker cross-slip of screw dislocations and climb of edge dislocations, leading to higher dislocation density in the alloy, as shown in Figs. 4a and c. When deformation temperature increases to 1150 ℃, enhanced thermal activation can activate slip systems that are otherwise unfavorable for deformation, forming a few subgrain boundaries, as shown in Fig. 4d. These subgrains become more active under stress and thermal activation, and fine subgrains can transform into subgrains with larger misorientation angles, causing the dynamic recrystallization mechanism to shift from discontinuous to continuous dynamic recrystallization. Additionally, increased deformation temperature enhances the migration capability of recrystallized grain boundaries and accelerates dislocation annihilation, thereby reducing dislocation density. Consequently, higher deformation temperatures result in lower dislocation density, clearer and sharper grain boundaries, and larger dynamic recrystallization grain sizes.

2.2 Microstructural Characteristics and Static Recrystallization Mechanism After Solution Annealing

The longitudinal section microstructure of the cold-rolled tube before solution treatment is shown in [Figure 5: see original paper]. The grains were elongated along the rolling direction, characteristic of cold deformation. [Figure 6: see original paper] shows the microstructures of specimens after various solution treatments. At a solution temperature of 1100 ℃, numerous fine equiaxed grains appeared, but the grain structure was quite inhomogeneous. As temperature increased, grains grew overall and became more uniform. Additionally, grains gradually increased in size with prolonged solution time. Notably, at 1180 ℃, recrystallization was completed within a very short time, and with increasing time, the alloy only underwent grain growth.

[Figure 7: see original paper] presents TEM observations of the cold-rolled and partially solution-treated specimens. The cold-rolled specimen contained numerous dislocation networks and deformation laths. As solution temperature increased (Figs. 7b-d) or solution time extended (Figs. 7e, c, f), recrystallization became more complete and dislocation density gradually decreased. Moreover, several high-angle grain boundaries exhibited certain dislocation density differences on both sides.

The driving force for static recrystallization is the mechanical stored energy of deformed metals and alloys; obtaining this stored energy satisfies the thermodynamic conditions for recovery and recrystallization. Specimens cut from the same tube had no significant difference in deformation amount or dislocation density. Higher annealing temperatures resulted in faster static recrystallization rates. Therefore, increased annealing temperature and extended annealing time both facilitate the release of deformation stored energy within the alloy, promoting grain growth and homogenization to restore the uniform microstructure prior to cold rolling. Similar to dynamic recrystallization, static recrystallization also occurs due to dislocation density differences across original high-angle grain boundaries caused by cold rolling, with strain-induced grain boundary migration serving as the primary recrystallization mechanism.

2.3 Step Grain Boundaries and Formation Mechanism During Recrystallization

[Figure 8: see original paper] and 9 show step grain boundary phenomena during dynamic and static recrystallization processes. In both stages, step grain boundaries exhibit different angles and heights; particularly during solution treatment, right-angle steps approach or exceed 0.5 m. Further observation reveals no phase formation at step corners, only some dislocations, which differs significantly from serrated grain boundaries with phase segregation reported in previous work [13~19]. Additionally, no twins were observed at the steps, ruling out the possibility of partial twinning.

According to fundamental materials science theory, crystal surfaces develop perfect low-energy planes (low-index planes) during near-equilibrium growth. Actual hot working processes are essentially non-equilibrium, causing crystal surfaces to deviate from these low-energy planes. Vicinal planes that slightly deviate from low-energy planes typically occupy most of the surface. These vicinal planes undergo faceting, where some atoms transfer from their original positions to other locations, forming new-oriented facets (as shown in [Figure 10: see original paper]), ultimately reducing surface energy. Such surfaces contain terraces, steps, and kinks, where terraces are low-energy, close-packed low-index planes, while steps and kinks are used to achieve deviation from low-index planes to ensure more surface area consists of low-energy planes. However, grain boundaries within polycrystals are similar yet different from free surfaces, because the lattices of two adjacent grains at the boundary undergo rigid offset and cannot directly form steps like free surfaces.

According to coincidence-site lattice (CSL) theory, grain boundary planes must intersect the most close-packed or close-packed planes of the CSL to maximize coincident sites or minimize repeat periods, thereby reducing grain boundary core energy and elastic strain energy. However, if the grain boundary plane orientation does not align with close-packed plane directions, the boundary decomposes into step-like grain boundaries composed of less close-packed planes [11]. This decomposition phenomenon can effectively improve the high-energy state of deformed grains.

The nickel-based alloy studied in this work has an fcc structure, where close-packed {111} planes have the lowest surface energy. If the external surface of a grain boundary is at an angle to the low-energy plane, the grain boundary external surface becomes stepped to maintain a low-energy surface state. Gleiter [24] found that in fcc materials, each {111} plane of a grain interacts with the grain boundary to form a step. Since each grain can typically induce four series of steps (four different {111} planes), up to eight different series of steps can exist simultaneously between adjacent grains. However, not all steps are visible; only those formed when low-energy planes are approximately parallel to the grain boundary exhibit strong contrast. If the deviation between low-energy planes and the grain boundary is large, the step morphology is not obvious. [Figure 9: see original paper] also shows that steps have different angles and heights, indicating these steps may be formed by multiple different low-energy planes and different numbers of low-energy plane layers. Crystallographically, different crystal plane families have different interplanar angles, so steps composed of different low-energy planes also have different angles. Additionally, steps constructed from different numbers of atomic layers of low-energy planes have varying heights.

Real grain boundaries are typically not planar but contain steps. Grain boundary steps are closely related to grain boundary dislocations; the core of grain boundary dislocations is typically the step present at the grain boundary [11], though the nucleation nature remains unclear. Only grain boundaries composed entirely of coincident sites have no steps or dislocations. The appearance of step grain boundaries indicates the crystal still has high dislocation content and large grain misorientation, meaning recrystallization is incomplete. Obviously, more coincident sites at grain boundaries result in less atomic arrangement distortion and lower grain boundary energy. Grain boundary dislocations are special defects that adjust interface energy, possessing a special Burgers vector **b**. Inserting a dislocation with the corresponding Burgers vector into a grain boundary changes the structure only in the vicinity of the dislocation, adding only a grain boundary step to give the interface more coincident sites, as shown in [Figure 11: see original paper]. As seen in the figure, different angles and relative positions between the grain boundary dislocation and grain boundary plane produce steps with different angles and heights.

At step grain boundaries deviating from close-packed planes, surface atoms have higher energy states and residual bonding bonds, making them susceptible to adsorption of foreign atoms and causing surface energy reduction. Driven by this force, atoms or atom clusters detach from the receding grain, move along steps across a certain thickness of grain boundary, while the growing grain absorbs an equivalent number of atoms from the boundary. This emission and absorption process is the microscopic mechanism of grain boundary migration [25,26]. This process causes step grain boundary movement and creates unstable material flow near the boundary, leading to grain growth or elimination, or even rearrangement. It should be noted that close-packed terrace structures are tightly arranged and unlikely to serve as atomic diffusion channels, while steps deviating from close-packed planes can. Therefore, the growth rate is maximum in the direction perpendicular to steps, causing lateral migration of stepped grain boundaries [27]. When steps sweep across the entire grain boundary, the whole boundary moves forward. However, step movement is affected by pinning from inherent defects on the grain boundary, which slows boundary movement during recrystallization. Nevertheless, this pinning enables some atomic-level fine steps [28] to be pinned and then merged by other moving steps to form micrometer-level steps shown in [Figure 8: see original paper] and 9. During actual recrystallization, grain boundaries can still migrate rapidly because grain boundary dislocation-step continuous nucleation and expansion, along with stored energy differences between recrystallized and original regions, dominate the boundary migration process. Only when the boundary enters a completely recrystallized grain interior does migration cease.

Additionally, as shown in [Figure 9: see original paper]d, step grain boundaries also exist during grain growth alone. [Figure 12: see original paper] shows TEM observations of a specimen after standard solution treatment and aging. Step grain boundaries persist even after the annealing recrystallization process is complete, indicating that after full recrystallization, step grain boundaries remain partially to reduce interface energy and continue promoting subsequent grain growth.

3 Conclusions

(1) Dynamic recrystallization during hot deformation of the nickel-based alloy for ultra-supercritical power plants is dominated by discontinuous dynamic recrystallization nucleated via grain boundary bulging, while static recrystallization during solution annealing of cold-rolled tubes is primarily governed by strain-induced grain boundary migration.

(2) Step grain boundaries with different morphologies formed during dynamic and static recrystallization serve to deviate surfaces from low-index planes, ensuring more grain boundary interfaces are low-energy close-packed planes, thereby reducing grain boundary energy. Their morphology depends on the crystallographic relationship of the grain boundary plane and the Burgers vector of grain boundary dislocations. The presence of step grain boundaries can also promote grain boundary migration and accelerate recrystallization. Moreover, after complete recrystallization, step grain boundaries remain partially in the alloy to reduce interfacial energy and continue promoting subsequent grain growth.

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