Effects of W and Re on Deformation and Recrystallization of Solution-Treated Nickel-Based Single-Crystal Superalloys: Postprint

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Abstract

Nickel-based single crystal superalloys were subjected to solution treatment, followed by deformation introduced via Brinell hardness tester indentation and shot peening, respectively. Recrystallization was subsequently observed after heat treatment to investigate the influence of W and Re elements on the deformation and recrystallization behavior of solution-treated nickel-based single crystal superalloys. The results demonstrate that the addition of W and Re leads to increased dislocation density and more extensive dislocation tangles after deformation, delayed recrystallization nucleation time (i.e., prolonged recrystallization incubation period), and significantly reduced recrystallization depth. Following shot peening, the surface microhardness of the single crystal superalloy increases substantially; with the addition of W and Re, the deformation depth decreases, the maximum recrystallization grain boundary migration rate is lowered, and along the direction of recrystallization depth, the variation in average grain boundary migration rate aligns with the trend of microhardness variation.

Full Text

Effect of W and Re on Deformation and Recrystallization of Solution Heat Treated Ni-Based Single Crystal Superalloys

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Abstract

Ni-based single crystal superalloys were subjected to deformation via Brinell indentation and shot peening after solution heat treatment, followed by heat treatment to observe recrystallization. The influence of tungsten (W) and rhenium (Re) on deformation and recrystallization behavior in solution-treated Ni-based single crystal superalloys was investigated. Results demonstrate that the addition of W and Re increases dislocation density and promotes dislocation tangling after deformation. This addition delays recrystallization nucleation, thereby extending the incubation period, and significantly reduces recrystallization depth. Following shot peening, surface micro-hardness increases substantially. With W and Re addition, the depth of the deformation zone decreases, and the maximum grain boundary migration rate is reduced. Moreover, along the recrystallization depth direction, the variation in average grain boundary migration rate correlates with the trend of micro-hardness changes.

Keywords: Ni-based single crystal superalloy, recrystallization, dislocation, micro-hardness, W, Re

Introduction

Superalloys exhibit high-temperature strength, excellent oxidation and hot corrosion resistance, superior creep and fatigue resistance, and good long-term microstructural stability. These materials are widely used in aerospace, energy, nuclear industries, and petrochemical sectors as critical structural materials essential for national defense and economic development. However, grain boundaries represent weak locations in materials at elevated temperatures, often leading to component failure. To meet the demands of increasing turbine inlet temperatures in high thrust-to-weight ratio aeroengines, directionally solidified columnar-grained and single crystal superalloys were developed, eliminating transverse grain boundaries or all grain boundaries entirely and substantially improving the temperature capability of superalloy components. Single crystal superalloys have found extensive applications in advanced aeroengines and industrial gas turbines, with their manufacturing and application capabilities serving as an important indicator of a nation' s materials development level.

The production of single crystal superalloy components involves complex processes including casting, shell removal, shot blasting, grinding, machining, heat treatment, and inspection. Throughout these intricate procedures, surface or localized deformation is difficult to avoid. When single crystal castings deform, recrystallization is readily induced during subsequent high-temperature solution treatment or service exposure. Recrystallization reintroduces transverse grain boundaries, causing a dramatic deterioration in the mechanical properties of single crystal superalloy components and representing a significant challenge in their application.

Consequently, researchers have conducted extensive studies on recrystallization in single crystal superalloys, primarily focusing on the effects of heat treatment parameters (temperature, time, and atmosphere), deformation modes and strain levels, and second-phase particles. However, investigations into the influence of alloy composition—an intrinsic property of the material—have been relatively limited. Minor carbon additions have been shown to hinder recrystallization through carbide formation, and increased contents of γ' -forming elements Al and Ti substantially suppress recrystallization. Nevertheless, the effects of solid solution strengthening elements on recrystallization in single crystal superalloys have not been reported. Therefore, this work selected W and Re, important solid solution strengthening elements in single crystal superalloys, to investigate their influence on deformation and recrystallization behavior.

Experimental Methods

The experimental materials were Ni-based single crystal superalloys. Master alloys without W and Re were first prepared by vacuum induction melting and cut into segments of approximately 3 kg. Single crystal superalloy bars (diameter 10 mm, length 200 mm) were then produced using a ZCD-10 high-gradient liquid metal cooling (LMC) directional solidification furnace, with W and Re added during the single crystal growth process. The nominal chemical compositions are listed in , with the alloys designated as DD00 (without W and Re) and DD0WR (with W and Re). The bars grew along the <001> crystallographic direction.

Discs measuring 10 mm in diameter and 5 mm in thickness were cut from the single crystal bars by wire electrical discharge machining. These specimens underwent solution heat treatment followed by water quenching to minimize the influence of γ/γ' eutectics and non-uniform γ' phases on recrystallization. The solution treatment regimes were 1250 °C for 2 h + 1300 °C for 2 h + 1310

°C for 4 h, water-cooled (for DD00) and 1250 °C for 3 h + 1330 °C for 10 h, water-cooled (for DD0WR).

The (001) surfaces of the solution-treated and quenched specimens were ground and then subjected to either Brinell indentation or shot peening. Brinell indentation was performed using a 500 kg load with a dwell time of 13 s. Shot peening conditions were as follows: shot diameter $ $75 m, average pressure 0.4 MPa, and peening duration 5 min. Some indented specimens were heat treated at 1310 °C for 4 h, while shot-peened specimens were heat treated at 1300, 1310, and 1330 °C for 4 h followed by air cooling. Additional specimens were heated at 1310 °C for various durations (0–300 s) and rapidly water-quenched to preserve transient microstructures.

All specimens were sectioned by wire electrical discharge machining: indented samples were cut along the indentation diameter, while shot-peened samples were sectioned perpendicular to the peened surface. Cross-sections were mechanically ground, polished, and etched for microstructural observation using an Axiovert200MAT optical microscope (OM) and Hitachi S-3400N scanning electron microscope (SEM). Recrystallization depth was measured as defined in reference [20]. The etchant consisted of 4 g $CuSO_4$ + 80 mL HCl + 20 mL C_2H_5OH.

Micro-hardness profiles from the shot-peened surface to the interior were measured on polished cross-sections using a Leco M-400-G automatic micro-hardness tester with a 50 g load and 13 s dwell time.

Thin foil specimens approximately 500 m thick were cut from indented samples and samples after short-time heat treatment at 1310 °C, parallel to the indentation plane. These foils were carefully ground to below 50 m, and 3 mm diameter discs were punched. Transmission electron microscopy (TEM) specimens were prepared by twin-jet electropolishing using a solution of 10% perchloric acid and 90% ethanol (by volume). Microstructural observations were conducted on a Tecnai 20 TEM.

Results

2.1 Recrystallization Microstructure After Heat Treatment

[Figure 1: see original paper] shows the γ' phase morphology in both alloys after solution treatment and quenching, representing the initial microstructure prior to recrystallization treatment. In the W- and Re-free DD00 alloy, the γ' phase exhibited a nearly cubic morphology with rounded corners and an average size of approximately 120 nm. In contrast, the DD0WR alloy containing W and Re showed γ' particles with a more perfect cubic morphology and a smaller size of about 80 nm.

[Figure 2: see original paper] presents the recrystallization microstructures after indentation and subsequent heat treatment at 1310 °C for 4 h. The surface matrix experienced oxidation-induced loss of Cr and Al elements, appearing lighter after etching under OM. The DD00 sample showed near-complete disappearance of the single crystal structure, with the specimen composed of several recrystallized grains of enormous size reaching millimeter scale [Figure 2a: see original paper]. The DD0WR alloy developed an ellipsoidal recrystallized region around the indentation with grain sizes of several hundred micrometers and a maximum recrystallization depth of approximately 1.1 mm [Figure 2b: see original paper]. This demonstrates that W and Re addition effectively suppresses recrystallization growth in single crystal superalloys.

[Figure 3: see original paper] shows the recrystallization microstructures after shot peening and heat treatment at 1310 °C for 4 h. The black holes represent high-temperature solution pores, whose formation mechanism is detailed in reference [21]. The DD00 alloy exhibited significantly larger recrystallized grain sizes and depths compared to the DD0WR alloy. The DD0WR alloy maintained relatively uniform grain sizes and generated numerous annealing twins [Figure 3b: see original paper].

Statistical analysis of maximum and mean recrystallization depths after shot peening and heat treatment at 1300, 1310, and 1330 °C for 4 h is presented in [Figure 4: see original paper]. The DD00 alloy showed substantially greater recrystallization depth than DD0WR, with maximum depth values reaching 2–5 times those of DD0WR [Figure 4a: see original paper]. The difference became more pronounced with increasing heat treatment temperature, indicating rapid recrystallization grain growth at certain locations in DD00. While maximum depth represents localized recrystallization, mean depth provides more global significance. Similarly, [Figure 4b: see original paper] reveals that the mean recrystallization depth of DD00 was markedly larger than that of DD0WR, with greater increases at higher temperatures.

2.2 Recrystallization During Short-Time Heat Treatment

To understand the effects of W and Re on recrystallization formation and development, both alloys were deformed by indentation and shot peening, then subjected to short-time heat treatment at 1310 °C for 10–300 s followed by water quenching to preserve transient microstructures.

[Figure 5: see original paper] shows the microstructures after indentation and short-time heat treatment. After 60 s at 1310 °C, DD00 developed recrystallized grains along the curved region beneath the indentation [Figure 5a: see original paper] with grain sizes of 100–200 m, while DD0WR showed no recrystallization [Figure 5b: see original paper]. At 120 s, the recrystallized region in DD00 grew rapidly into an ellipsoidal shape [Figure 5c: see original paper] with grain sizes reaching millimeter scale. DD0WR also recrystallized [Figure 5d: see original paper] but with significantly smaller depth and only a few grains near the indentation edge.

[Figure 6: see original paper] presents statistical analysis of recrystallization depth and grain number after indentation and heat treatment at 1310 °C. Both alloys exhibited an incubation period: approximately 10 s for DD00 and 60 s for DD0WR, indicating that W and Re addition delays recrystallization nucleation. Recrystallization depth increased with heating time in both alloys, but the slope was smaller for DD0WR, suggesting slower recrystallization growth. [Figure 6b: see original paper] shows the variation in recrystallized grain number with heat treatment time (excluding twins). While DD00 exhibited approximately linear grain number increase during DD0WR' s incubation period—indicating nucleation dominance—DD0WR's grain number began increasing after the incubation period and continued growing throughout the experiment. In contrast, DD00 showed a rapid decrease in grain number after 120 s due to abnormal grain growth consuming numerous grains [Figure 5c: see original paper].

[Figure 7: see original paper] shows microstructures after shot peening and short-time heat treatment at 1310 °C. After 60 s, both alloys had passed the incubation period and recrystallized. DD00 developed a continuous recrystallized layer at the shot-peened edge with substantial depth, while DD0WR showed very small recrystallization depth [Figure 7b: see original paper]. After 120 s, DD00 recrystallization continued growing, whereas DD0WR depth remained minimal.

[Figure 8: see original paper] illustrates the variation in mean recrystallization depth with time after shot peening and heat treatment at 1310 °C. The incubation period was approximately 10 s for DD00 and 30 s for DD0WR, demonstrating that W and Re extend the recrystallization incubation period in shot-peened single crystal superalloys. After incubation, recrystallization depth increased rapidly with time, with DD00 showing significantly greater depth than DD0WR. During the initial growth stage (30–120 s), the growth rate of mean recrystallization depth in DD00 was markedly higher than in DD0WR.

Recrystallization in shot-peened specimens nucleated at the surface and expanded inward and laterally. No secondary nucleation occurred along the depth direction; growth primarily involved surface-nucleated grains, consistent with literature reports. Since secondary nucleation was absent, recrystallization depth equaled grain boundary migration distance. Therefore, the increase in mean recrystallization depth per unit time (slope in [Figure 8: see original paper]) corresponds to the mean grain boundary migration velocity.

[Figure 9: see original paper] shows the variation in mean recrystallization grain boundary migration velocity with time after shot peening and heat treatment at 1310 °C. Both alloys exhibited an initial increase followed by a decrease in migration velocity, indicating a maximum value. However, two differences were observed: (1) DD00 showed a higher maximum migration velocity, and (2) the time to reach maximum velocity differed—60 s for DD00 versus 120 s for DD0WR, indicating delayed attainment of maximum velocity in the W- and Re-containing alloy.

[Figure 10: see original paper] presents the relationship between mean recrystallization grain boundary migration velocity and mean recrystallization depth (equal to distance from the shot-peened surface). Within approximately 35 m from the surface, DD00 migration velocity continued increasing until reaching its maximum, whereas DD0WR migration velocity first increased then decreased, with its maximum occurring closer to the surface.

2.3 Micro-Hardness

Micro-hardness characterizes material strength, and differences in micro-hardness after deformation can reflect strength increases resulting from deformation. [Figure 11: see original paper] shows the micro-hardness profiles along the cross-section after shot peening. The DD0WR alloy exhibited higher micro-hardness than DD00. After shot peening, both alloys showed significant surface hardness increases, but the depth of hardness increase (Dd_2) in DD0WR was smaller than in DD00 (Dd_1), indicating a shallower deformation zone.

2.4 TEM Observations

[Figure 12: see original paper] shows TEM images after indentation deformation. In DD00, distinct slip bands formed with relatively high dislocation density within the bands but sparse dislocations between them [Figure 12a: see original paper]. In contrast, DD0WR exhibited parallel dislocations with numerous additional dislocations tangled between them, resulting in high overall dislocation density [Figure 12b: see original paper].

[Figure 13: see original paper] shows dislocation configurations after indentation and short-time heat treatment at 1310 °C for 60 s. In DD00, the approximately circular particles are γ' phase. Dislocations exhibited partial bending at γ' particles (arrows), suggesting climb-mediated bypassing. The relatively dispersed dislocation distribution and shorter dislocation line lengths facilitated rapid recovery. In DD0WR, numerous dislocation tangles persisted after short-time heat treatment [Figure 13b: see original paper]. Compared to [Figure 12b: see original paper], the tangle nodes were larger, with increasing dislocation spacing from the node center outward, indicating partial tangle loosening. Arc-shaped dislocation configurations were also observed, with curvature dimensions comparable to γ'particle size, suggesting operation of the Orowan bypassing mechanism during deformation. The absence of dislocation loops indicates their elimination via thermally activated climb.

Discussion

3.1 Effect of W and Re on Deformation of Single Crystal Superalloys

For single crystal superalloys without minor elements such as C and B, strengthening mechanisms primarily include solid solution strengthening and γ' particle strengthening. Although the solution treatment quenching time was extremely short, γ' phase precipitation was inevitable. W and Re addition reduced γ' particle size while maintaining similar volume fractions. Literature reports indicate that within the small particle size regime (<500 nm), alloy strength decreases with decreasing γ' size. However, micro-hardness measurements showed higher hardness in DD0WR, suggesting that the strength increase originates from solid solution strengthening by W and Re. During deformation, W and Re impede dislocation motion, enhancing interactions between solute atoms and dislocations as well as between dislocations themselves, thereby increasing dislocation density and post-deformation micro-hardness. The higher strength of DD0WR results in lower deformability under identical loading conditions, producing a shallower deformation zone and consequently reduced recrystallization depth.

3.2 Effect of W and Re on Recrystallization Nucleation

Both indentation-induced localized deformation and shot peening-induced uniform deformation resulted in significantly longer recrystallization incubation periods in DD0WR compared to DD00, demonstrating that W and Re extend the recovery stage and delay recrystallization nucleation. The incubation period involves microstructural changes including vacancy annihilation and dislocation rearrangement and elimination. The high dislocation density and extensive dislocation tangles formed in DD0WR during deformation are difficult to loosen through dislocation motion during subsequent heat treatment. These tangles also impede other dislocation movements, retarding recovery. [Figure 13c: see original paper] shows that deformation-generated dislocations in DD0WR were longer, with individual dislocations wrapping around numerous γ' particles, creating substantial Orowan bypassing resistance. Consequently, dislocation elimination is difficult in DD0WR, resulting in a prolonged recrystallization incubation period. Additionally, slower dislocation motion in DD0WR may be associated with solute element segregation at dislocations.

3.3 Effect of W and Re on Recrystallization Grain Growth

Recrystallization grain growth manifests as grain boundary migration. After nucleation, grain boundaries migrate and recrystallized grains grow rapidly with increasing heat treatment time, but the growth rate (boundary migration rate) in DD0WR is significantly lower than in DD00. The grain boundary migration velocity can be expressed as:

$$v = M \cdot P$$

where v is the migration velocity, M is the grain boundary mobility, and P is the driving pressure. Studies indicate that increased solute atom content reduces grain boundary mobility M. Therefore, the addition of 6% W and 4% Re is expected to decrease M. The driving pressure P equals the difference between the driving force Pd (primarily from stored deformation energy) and the migration resistance. W and Re increase dislocation density in DD0WR, resulting in higher driving force Pd. However, migration resistance includes solute drag force Psol and particle Zener pinning force Pz. W and Re have large atomic radii (1.25 and 1.22 times that of Ni, respectively) and high diffusion activation energies. Their enrichment at dislocations and γ/γ' interfaces increases Psol. Since Pz is inversely proportional to particle radius, the smaller γ' particles in DD0WR [Figure 1: see original paper] result in larger Pz. Although W and Re increase Pd in DD0WR, the combined effects of increased Psol and Pz and decreased M reduce the grain boundary migration velocity and recrystallization depth.

Both alloys exhibited a maximum in mean recrystallization grain boundary migration velocity, with velocity varying significantly along the recrystallization depth direction. Comparing [Figure 10: see original paper] and [Figure 11: see original paper] reveals that the maximum micro-hardness in DD0WR occurred at 15-20 m from the surface, while its maximum mean grain boundary migration velocity appeared at approximately 15 m. For DD00, maximum micro-hardness occurred at 15-25 m, with maximum migration velocity at 25-35 m. The regions of maximum micro-hardness and maximum migration velocity essentially coincide. Increased micro-hardness indicates higher dislocation density and greater driving force for grain boundary migration, leading to enhanced migration velocity. Although the maximum micro-hardness in DD0WR occurred closer to the surface, the longer incubation period and slower migration velocity delayed the appearance of the maximum mean grain boundary migration velocity.

Conclusions

(1) The addition of W and Re reduces recrystallization depth in single crystal superalloys after both localized indentation deformation and uniform shot peening deformation, demonstrating that W and Re suppress recrystallization.

(2) W and Re extend the recrystallization incubation period in single crystal superalloys, which is attributed to slower dislocation elimination and prolonged recovery.

(3) W and Re increase micro-hardness, thereby reducing the deformation zone depth after shot peening and ultimately decreasing recrystallization depth.

(4) W and Re addition reduces the mean recrystallization grain boundary migration velocity in single crystal superalloys. In shot-peened specimens, the variation in mean grain boundary migration velocity along the recrystallization depth direction correlates with micro-hardness changes.

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