Effect of Transverse Pre-compression on Creep Behavior of [011]-Oriented Nickel-Based Single Crystal Superalloy (Postprint)

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Abstract

Through creep property testing and microstructural observation, the influence of transverse pre-compression on the tensile creep behavior of [011] oriented nickel-based single crystal superalloy at 1040 ℃ and 137 MPa was investigated. The results indicate that after pre-compressing a [011] oriented nickel-based single crystal superalloy along the [100] direction for 38 h at 1040 ℃ and 180 MPa, a P-type γ' rafted structure generally parallel to the compression stress axis was formed. The different distributions of strain energy density and lattice strains on various crystal planes during pre-compression are the primary causes for the rafting of the γ' phase. Dislocation slip in the matrix channels is the main deformation mechanism for [011] oriented alloys with and without pre-compression during the steady-state creep stage, with more extensive dislocation climb observed in the pre-compressed alloy. Transverse pre-compression significantly enhanced the creep resistance of [011] oriented alloy under the conditions of 1040 ℃ and 137 MPa. The elimination of "gable-type" matrix channels in the alloy, the smaller γ matrix width during the steady-state creep stage, the micro-bottleneck-shaped γ matrix channels formed by lateral connection of rafted structures, the "labyrinth-like" microstructure, and the activation of multiple slip systems all contribute to restricting dislocation motion and thereby improving the creep resistance of the pre-compressed alloy.

Full Text

1. Experimental Methods

Single crystal bars of a nickel-based superalloy with composition Ni-9.0Cr-5.0W-5.5Al-4.5Co-1.7Ti (mass fraction, %) were prepared along the [011] orientation using a seed crystal method in a high-temperature gradient vacuum directional solidification furnace. The deviation of the single crystal bars from the [011] orientation was determined to be 4° by Laue back-reflection. The bars were subsequently heat-treated in a box resistance furnace using the following process: 1250 °C for 4 h, air-cooled, followed by 870 °C for 32 h, air-cooled. Two parallel loading surfaces were cut along the (100) crystal plane from the fully heat-treated single crystal bars, as shown in [Figure 1: see original paper]a. The bars were placed in a custom-built reverse-tension device and subjected to high-temperature pre-compression at 1040 °C and 180 MPa along the [100] direction for 38 h to ensure transformation of the g’ phase into a rafted structure. For convenience, the [011] oriented alloy after heat treatment is designated as Alloy 1, and the pre-compressed [011] oriented alloy as Alloy 2.

Creep test specimens were machined from Alloys 1 and 2 along the (100) and (011) crystal planes into plate-shaped tensile creep samples with a gauge length of 11.0 mm and a cross-section of 4.5 mm × 2.5 mm in the gauge portion. The wide surface of the specimens was the (100) crystal plane, with a schematic shown in [Figure 1: see original paper]b. After surface mechanical polishing, the creep specimens were placed in a GWT504 high-temperature creep testing machine for tensile creep performance testing along the [011] orientation at 1040 °C and 137 MPa, with creep curves recorded. Specimens of Alloys 1 and 2 before and after creep were mechanically ground and polished, then etched using either 100 mL HCl + 80 mL H_2O + 20 g $CuSO_4$ or 10 g $(NH_4)_2SO_4$ + 10 g $C_6H_8O_7$ + 1200 mL H_2O electrolyte. Microstructural morphologies on different crystal planes were observed using an S-3400N scanning electron microscope (SEM) to construct the spatial distribution of g’ /g phases. The dimensions of g’ phase and g channels in different alloy states (three SEM images per state) were measured and statistically averaged to analyze the effect of dimensional changes on creep behavior. Specimens crept to the steady-state stage were fabricated into 3 mm diameter discs approximately 50 m thick. Thin foils were prepared using an MTP-1A twin-jet electropolishing apparatus at temperatures below -20 °C with a solution of 10% $HClO_4$ + 90% C_2H_5OH (volume fraction), and microstructural observations were performed using a TECNAI-20 transmission electron microscope (TEM) to investigate deformation mechanisms.

2.1 Microstructure of the Alloy

Orthogonal hexahedra were sectioned from Alloy 1 along the (100), (010), and (011) crystal planes, with the (010) plane obtained by cutting the (100) plane at 45° to the vertical direction. [Figure 2: see original paper]a shows a schematic of the crystal planes in a single crystal cell, while [Figure 2: see original paper]b-d present the microstructures on the (010), (100), and (011) planes, respectively. On the (010) plane, cuboidal g’phase is coherently embedded in the g matrix and regularly arranged along the [001] and [100] directions. On the (100) plane, the g’ phase maintains a cubic morphology, characterized by corners aligned parallel to the [011] direction and edges arranged along the [010] and [001] directions. On the (011) plane, the g’ phase exhibits a rectangular shape along the [011] orientation. These observations indicate that after full heat treatment, the

[011] oriented single crystal alloy possesses a spatial microstructure of cuboidal g' phase coherently embedded in the g matrix and regularly arranged along <100> orientations. [Figure 2: see original paper]e schematically illustrates the g' /g phase arrangement in Alloy 1, where (001)gr and (010)gr represent "roof-type" matrix channels parallel to the (001) and (010) crystal planes, respectively, while (100)gg denotes "gable-type" matrix channels parallel to the (100) crystal plane.

2.2 Microstructure After Pre-compression

[Figure 3: see original paper]a shows a schematic of a unit cell under compressive stress along the [100] direction in Alloy 2. [Figure 3: see original paper]b presents the microstructure on the (100) plane, where g' phase particles are primarily distributed as ellipsoidal or string-like shapes with an arrangement similar to that in [Figure 2: see original paper]c: the "corners" of cuboidal g' phase align parallel to the [011] direction, with edges regularly arranged at 45° to the [011] orientation, though the g' phase edges become spheroidized after pre-compression. Notably, many g' phase particles connect along the [001] and [010] directions, as indicated by arrows 1 and 2 in [Figure 3: see original paper]b. Similar morphologies are observed on the (011) and (011) planes in Alloy 2, shown in [Figure 3: see original paper]c, where the g' phase has transformed into wavy rafted structures generally parallel to the compressive stress axis [100]. Based on these three crystal plane microstructures, the spatial morphology of rafted g' phase can be constructed, as shown in [Figure 3: see original paper]d, where arrows 6 and 7 indicate that variations in cutting plane position may cause non-uniform g' raft width display on the same plane, consistent with [Figure 3: see original paper]c. Compared with Alloy 1 ([Figure 2: see original paper]e), Alloy 2 exhibits elimination of the "gable-type" channel (100)gg, while retaining two "roof-type" matrix channels (001)gr and (010)gr. Micro-bottleneck-like matrix channels exist between g' rafts, as shown by arrows 3 and 4 in [Figure 3: see original paper]c. Lateral connections perpendicular to the [100] direction appear between rafted g' phases, indicated by arrow 5, which is related to lateral growth of g' rafts, misalignment between the compressive stress axis and [100] orientation, and the alloy' s orientation deviation. This is illustrated in the dashed box region of [Figure 3: see original paper]d, showing labyrinth-like microstructures. This labyrinthine microstructure alters the continuously open "roof-type" channel configuration ([Figure 2: see original paper]e), increases the obstacle density for dislocation motion, forces dislocations to continuously change direction during creep, increases the proportion of climb in dislocation motion, and enhances dislocation motion resistance, thereby improving creep resistance.

2.3 Effect of Pre-compression on Creep Properties

[Figure 4: see original paper] shows the tensile creep curves of Alloys 1 and 2 at 1040 °C and 137 MPa, with creep parameters listed in . Alloy 2 exhibits smaller initial strain and steady-state creep rate compared with Alloy 1, while its creep life is improved by approximately 123%. Pre-compression significantly enhances the creep resistance of the [011] oriented alloy.

2.4 Microstructural Evolution During Creep

Alloy 2 was tensile crept along the [011] orientation at 1040 °C and 137 MPa for 60 h. [Figure 5: see original paper]a shows a schematic of the unit cell during tensile creep and various crystal planes. [Figure 5: see original paper]b presents the microstructure on the (100) plane, which remains similar to that before creep ([Figure 3: see original paper]b), dominated by ellipsoidal and string-like morphologies, though with significantly increased dimensions. The g' phase further connects along the [001] and [010] directions, as indicated by arrows in [Figure 5: see original paper]b. Similar morphologies are observed on the (011) and (011) planes ([Figure 5: see original paper]c), indicating that the rafted g' phase morphology perpendicular to the tensile stress axis remains intact. Overall, Alloy 2 maintains a stable internal microstructure during tensile creep along the [011] direction, retaining a one-dimensional rafted morphology perpendicular to the tensile stress axis and preserving the labyrinthine microstructure. Ignoring g' /g phase dimensional changes, its spatial organization remains as schematically shown in [Figure 3: see original paper]d.

2.5 Evolution of g' /g Phase Dimensions

Microstructural evolution occurs in nickel-based single crystal superalloys during creep, causing changes in g' /g phase dimensions that significantly affect creep behavior. The microstructure within interdendritic and dendritic regions is not uniform. [Figure 6: see original paper]a shows the dendritic morphology of the [011] oriented alloy after heat treatment, where point A is located in the interdendritic region and point B in the dendritic region. [Figure 6: see original paper]a serves as a schematic for both regions to identify microstructures in different alloy states, though the contrast between interdendritic and dendritic regions becomes much less pronounced after pre-compression and tensile creep in Alloys 1 and 2 (figures omitted). [Figure 6: see original paper]b-e show microstructures in interdendritic regions on the (100) plane for different alloy states, while [Figure 6: see original paper]f-i show dendritic region microstructures: [Figure 6: see original paper]b and f for Alloy 1, [Figure 6: see original paper]c and g for Alloy 2, [Figure 6: see original paper]d and h for Alloy 1 after 40 h creep at 1040 °C and 137 MPa, and [Figure 6: see original paper]e and i for Alloy 2 after 60 h creep under the same conditions. Significant dimensional changes in g' /g phases occur during pre-compression or tensile creep, with measurement results summarized in .

2.6 Deformation Mechanisms During Creep

[Figure 7: see original paper] shows microstructures on the (100) plane for Alloys 1 and 2 crept to the steady-state stage at 1040 °C and 137 MPa, where double arrows indicate the tensile stress axis direction. [Figure 7: see original paper]a shows Alloy 1 after 40 h creep. No dislocation shearing of g’ phase is observed, indicating that steady-state strain in Alloy 1 originates from dislocation motion within the matrix. The inset in [Figure 7: see original paper]a, an enlarged view of the boxed region, reveals dislocation climb at g’ /g interfaces, though this is not prominent throughout the field of view. [Figure 7: see original paper]b shows the pre-compressed alloy after 60 h creep. The g’ phase in Alloy 2 maintains ellipsoidal and string-like morphologies on the (100) plane, with a few dislocations cutting into the g’ phase (arrow 1), likely related to stress concentration caused by dislocation pile-up at the labyrinthine structure. The inset in [Figure 7: see original paper]b shows dislocation bowing and climb between two g’ phases, with similar motion observed elsewhere (arrows 2 and 3). In summary, the deformation mechanisms for both alloys crept to the steady-state stage at 1040 °C and 137 MPa involve dislocation slip, with Alloy 2 exhibiting more extensive dislocation bowing and climb. Limited dislocation cutting of rafted structures is not the primary deformation mechanism for either alloy during steady-state creep, consistent with literature results, so this work considers only dislocation motion in the matrix.

3.1 Directional Diffusion of Elements During Pre-compression

During pre-compression at 1040 °C and 180 MPa, the g’ phase in the [011] oriented nickel-based single crystal superalloy undergoes rafting transformation ([Figure 3: see original paper]) due to directional diffusion of alloying elements. After full heat treatment, cuboidal g’ phase is coherently embedded in the g matrix (spatial relationship shown in [Figure 2: see original paper]e). The alloy possesses a negative lattice misfit δ, meaning a_g > a_g’ (where a is lattice constant). Consequently, without external stress, the g matrix experiences coherent compressive stress while cuboidal g’ phase experiences tensile stress, as schematically shown in [Figure 8: see original paper]a.

When compressive stress is applied along the [100] direction, the “roof-type” matrix channels (001)gr and (010)gr in the [011] oriented alloy become vertical channels, while the “gable-type” matrix channel (100)gg becomes a horizontal channel ([Figure 2: see original paper]e). In the stress-free state, elastic strain energy density in the g matrix distributes symmetrically along horizontal and vertical channels. Additionally, since the elastic modulus and strength of g matrix channels are lower than those of the g’ phase, their strain energy density is higher. Applied external stress alters the elastic strain energy density distribution between the two phases, inevitably changing the local equilibrium concentration of solute elements and resulting in directional coarsening of the g’ phase. In this study, external compressive stress causes the horizontal channel to experience tension parallel to the (100) plane, largely offsetting the misfit compressive stress, while vertical channels experience superposition of external compressive stress along [100] and misfit compressive stress, resulting in higher elastic strain energy density in vertical channels than in horizontal ones. Dislocations preferentially initiate and distribute in vertical channels. Since Cr and

Co elements in the g matrix have higher mobility, they first enter these dislocations to reduce elastic strain energy. The released energy drives diffusion of g' -forming elements (Al and Ti, etc.). Therefore, during compressive stress application, some Cr and Co elements in the g matrix diffuse along dislocations until saturating the dislocation regions. This elemental diffusion disrupts the original chemical equilibrium, and according to the equilibrium partitioning principle, a reverse diffusion flux of g' -forming elements (Al and Ti, etc.) occurs, leading to directional coarsening of the g' phase parallel to the compressive stress axis.

From another perspective, directional diffusion during pre-compression is related to lattice strain on different crystal planes of cuboidal g' phase and changes in elemental chemical potential. In the unstressed state, misfit stress at g' /g interfaces caused by lattice mismatch distributes uniformly across all crystal planes. This misfit stress-induced strain near the interface exhibits identical distribution patterns along all crystallographic directions for both g' and g phases, as shown in [Figure 8: see original paper]a. When compressive stress is applied along the [100] direction, boundaries of cuboidal g' phase along different crystallographic directions experience different types of lattice strain ([Figure 8: see original paper]b), which further affects stress distribution on different g' phase planes and changes in elemental chemical potential.

Notably, during compressive stress application at high temperature, dislocation motion dominates in the matrix, while the g' phase undergoes only elastic strain without dislocation penetration. Although dislocation slip in the g matrix can accelerate elemental diffusion through pipe diffusion, plastic deformation in matrix channels has negligible effect on elastic strain of cuboidal g' phase. [Figure 9: see original paper] schematically illustrates directional growth of g' phase and g' /g interface migration during compressive stress along [100] in the [011] oriented alloy. Defining boundaries of g' phase particles parallel to (001), (010), and (100) planes as (001)g' , (010)g' , and (100)g' , respectively, compressive stress along [100] generates compressive strain in (001)g' along the [100] direction, expelling Al and Ti atoms with larger atomic radii into nearby g matrix channels (001)gr, causing Al and Ti supersaturation and increased chemical potential at this location (region A in [Figure 9: see original paper]a). Similarly, Al and Ti elements in (010)g' are also expelled into nearby matrix channels (010)gr (figure omitted), raising the chemical potential of these elements.

According to generalized Hooke' s law, compressive strain on (010) and (001) planes of a g' particle under uniaxial compression necessarily generates expansive strain on its (100)g' plane, creating interstitials along [010] and [001] directions and increasing system strain energy, placing the system in a metastable state. When expelled g'-forming elements with larger atomic radii diffuse from vertical channels (001)gr and (010)gr into (100)gg and enter g' phase interstitials, the nearby g matrix phase transforms in-situ to g' phase while reducing system strain energy. This spontaneous process accompanies system energy reduction. This directional diffusion process continuously expels g' -forming elements from (001)g' and (010)g' planes through matrix channels into (100)g' , causing g' phase to grow continuously on the (100) plane along the [100] direction and the g' /g interface to migrate in this direction (vertical arrows in [Figure 9: see original paper]b) until adjacent g' particles connect to form P-type rafted structures parallel to the stress axis ([Figure 9: see original paper]c). During this process, g phase transformation causes its forming elements Cr and Co to precipitate and enrich in region B of [Figure 9: see original paper]a, creating supersaturation and increased chemical potential, which establishes a chemical potential gradient for Cr and Co between (001)gr and (100)gg, promoting their diffusion into matrix channel (001)gr. This widens the "roof-type"matrix channel and reduces g' phase dimension along [001] (horizontal arrows in [Figure 9: see original paper]b). Similarly, the other "roof-type" matrix channel (010)gr undergoes analogous widening and g' phase dimension reduction along [010] (figure omitted). This analysis demonstrates that lattice strain on different g' phase planes and resulting changes in elemental chemical potential are the driving forces for directional diffusion during pre-compression of [011] oriented alloy along [100].

3.2 Influence of Microstructure on Creep Behavior

Compared with Alloy 1 microstructure ([Figure 2: see original paper], [Figure 6: see original paper]b, and), Alloy 2 microstructure ([Figure 3: see original paper], [Figure 6: see original paper]c, and) shows significant changes that affect initial creep behavior. First, the (100)gg matrix channel disappears, eliminating dislocation motion within it and reducing initial creep rate. Second, (001)gr and (010)gr matrix channels remain but are frequently obstructed by g'phase growing laterally along [010] and [001] directions, causing channel narrowing or complete blockage (arrows 1 and 2 in [Figure 3: see original paper]b and dashed box in [Figure 3: see original paper]e). This labyrinthine structure forces dislocations to continuously change direction during motion, increasing motion resistance and reducing creep rate. Third, although Alloy 2 g matrix channel dimensions (~75 nm in interdendritic and ~70 nm in dendritic regions) are larger than those in Alloy 1 (~52 nm and ~49 nm in corresponding regions), Alloy 2 g' raft widths (~760 nm and ~650 nm) are substantially greater than Alloy 1 g' particle widths (~430 nm and ~390 nm), increasing dislocation climb resistance. Additionally, although Alloy 2 g matrix channels are wider than in Alloy 1, they are non-uniform, with lateral g' raft growth creating microscopic "bottlenecks" (arrows 1 and 2 in [Figure 6: see original paper]g) that also restrict dislocation motion. For these reasons, Alloy 2 microstructure is more effective at limiting dislocation motion, resulting in smaller initial strain and creep rate compared with Alloy 1 ([Figure 4: see original paper] and).

Alloy 2 exhibits lower creep rate during the mid-stage and longer creep life compared with Alloy 1. [Figure 10: see original paper] schematically illustrates Alloy 1 microstructure after 40 h creep at 1040 °C and 137 MPa based on literature. This differs markedly from Alloy 2 microstructure crept to 60 h under the same conditions ([Figure 3: see original paper]d). After steady-state creep, Alloy 2 (001)gr and (010)gr channel widths (~157 nm in interdendritic and ~125 nm in dendritic regions) are less than half those of Alloy 1 (010)gr (excluding (100)gg for reasons discussed below; (001)gr disappears) (~400 nm and ~290 nm), while its g' raft widths (~850 nm and ~750 nm) are significantly larger than Alloy 1 g' raft widths (~450 nm and ~320 nm). The pre-compressed [011] oriented alloy maintains stable microstructure after steady-state creep, retaining labyrinthine matrix channels ([Figure 3: see original paper]d and [Figure 5: see original paper]b).

Considering the common matrix channel (010)gr in both alloys during steady-state creep, the effect of these microstructural changes on creep behavior can be illustrated using the (111)$[\bar{1}01]$ slip system as an example ([Figure 11: see original paper]). As previously discussed, the primary deformation mechanism during steady-state creep for both alloys is dislocation slip in matrix channels, with more extensive dislocation bowing and climb in Alloy 2. [Figure 11: see original paper]a schematically shows dislocation motion in the (010)gr channel, where rafts 1 and 1' represent ordinary g' raft morphology that does not affect (010)gr openness, rafts 2 and 2' form microscopic bottlenecks due to lateral g' raft growth along [010], and rafts 3 and 3' completely connect laterally along [010], blocking the (010)gr channel and forming labyrinthine microstructure. AB, BC, and CD in [Figure 11: see original paper]a represent dislocation slip, bowing, and climb, respectively. After completing climb (CD), dislocations can continue slipping in matrix channels (DE). When encountering raft 3(3') in the (010)gr channel, dislocations can pile up, and newly arriving dislocations react with piled-up dislocations and change direction, as shown by segments EF and EG.

The Orowan stress τ_{or} required to overcome dislocation motion during bowing can be expressed as:

$$\tau_{or} = (\mu b)/L$$

where is the shear modulus, b is the Burgers vector magnitude, and L is the distance between g' phases along <110> directions. Compared with Alloy 1 crept to steady-state at 1040 °C and 137 MPa, Alloy 2 exhibits narrower matrix channels (including retained micro-bottlenecks) after equivalent creep, which retards dislocation bowing. Additionally, larger g' raft widths in Alloy 2 make dislocation climb more difficult, also contributing to reduced steady-state creep rate and improved creep resistance. In this sense, Alloy 2 labyrinthine structure forces dislocations to climb, while Alloy 1 open "roof-type" channel (010)gr and open "gable-type" channel (100)gg present fewer obstacles to dislocation slip, resulting in less climb activity (the climb observed in [Figure 7: see original paper]a may originate from g' phase misalignment or irregular shapes).

3.3 Effect of Slip System Activation

During tensile creep of [011] oriented alloy along [011], four equivalent slip systems with identical Schmid factors (0.408) can be activated (), considering only octahedral slip systems under high-temperature creep conditions. Due to significant cancellation between misfit compressive stress and applied tensile stress in vertical "gable-type" channels (parallel to the tensile stress axis), the low effective stress cannot support dislocation motion. Consequently, dislocation motion during creep of [011] oriented alloy concentrates primarily in "roof-type" matrix channels. Dislocations in the $(111)[1\bar{1}0]$ and $(\bar{1}11)[101]$ slip systems mainly operate in (001)gr, while those in the $(111)[\bar{1}01]$ and $(\bar{1}11)[110]$ systems operate primarily in (010)gr. The fewer activated slip systems and resulting weaker strain hardening are important reasons for poor creep resistance in [011] oriented alloys.

In Alloy 1 crept to steady-state at 1040 °C and 137 MPa, (001)gr disappears ([Figure 10: see original paper]), restricting dislocation motion in the $(111)[1\bar{1}0]$ and $(\bar{1}11)[101]$ slip systems. Alloy strain primarily originates from dislocation motion in two equivalent slip systems within (010)gr. In contrast, the pre-compressed [011] oriented alloy retains both (001)gr and (010)gr after equivalent creep, enabling four effectively activated equivalent slip systems—more than in Alloy 1. This greater number of activated slip systems and resulting rapid strain hardening are also important reasons for Alloy 2' s lower steady-state creep rate ([Figure 4: see original paper]).

In addition to dislocation slip and climb, considering the alloy' s orientation deviation, dislocations may also undergo cross-slip and double cross-slip in matrix channels, as schematically shown in [Figure 11: see original paper]b. Screw dislocations can cross-slip from (111) to $(1\bar{1}1)$ plane and double cross-slip back to (111) plane, with curve HIJKLMNO representing this process. Compared with the non-pre-compressed alloy, the narrower matrix channels in Alloy 2 at steady-state restrict cross-slip and reduce steady-state creep rate.

Conclusions

(1) After pre-compression at 1040 °C and 180 MPa along the [100] direction for 38 h, the [011] oriented nickel-based single crystal superalloy forms P-type rafted structures generally parallel to the compressive stress axis, with lateral connections between g' rafts creating micro-bottleneck-like matrix channels and labyrinthine microstructures.

(2) During pre-compression, the driving force for P-type rafting is the higher/lower strain energy density in matrix channels parallel/perpendicular to the compressive stress axis. Boundaries of g' phase parallel/perpendicular to the stress axis experience compressive/expansive lattice strains that expel/capture g' -forming elements with larger atomic radii (Al and Ti, etc.), promoting directional diffusion and rafting of g' phase along directions normal to expansively strained crystal planes.

(3) Dislocation slip in matrix channels is the primary deformation mechanism during steady-state creep for both pre-compressed and non-pre-compressed [011] oriented alloys, with more extensive dislocation climb

occurring in the pre-compressed alloy.

(4) The bottleneck-like and narrow g matrix channels between rafted g' phases, labyrinthine microstructure, and activation of more slip systems than in the non-pre-compressed alloy are the main reasons for improved creep resistance of the pre-compressed [011] oriented alloy at 1040 °C and 137 MPa.

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