The dependence of stress and strain rate on the deformation behavior of a Ni-based single crystal superalloy at 1050°C

Qingqing Ding1 | Hongbin Bei1 | Lulu Li2 | Jie Ouyang2 | Xinbao Zhao1 | Xiao Wei1 | Ze Zhang1

1School of Materials Science and Engineering, Zhejiang University, Hangzhou, China
2Polytechnic Institute, Zhejiang University, Hangzhou, China

Correspondence
Assoc. Prof. Qingqing Ding, Prof. Hongbin Bei, and Academician Prof. Ze Zhang, School of Materials Science and Engineering, Zhejiang University, Hangzhou 310027, China. Email: qq_ding@zju.edu.cn, hbei2018@zju.edu.cn, and zezhang@zju.edu.cn

Funding Information
The Basic Science Center Program for Multiphase Media Evolution in Hypergravity of the National Natural Science Foundation of China, Grant/Award Number: 51988101; The Innovation Fund of the Zhejiang Kechuang New Materials Research Institute, Grant/Award Numbers: ZKN-20-P01, ZKN-20-201: The Fundamental Research Funds for the Central Universities, Grant/Award Number: 2020QNA4004: The Key R & D Project of Zhejiang Province, Grant/Award Number: 2020C01002; Natural Science Foundation of Zhejiang Province, Grant/Award Number: LQ20E01008; National Natural Science Foundation of China, Grant/Award Number: 91960201

Abstract
Ni-based single crystal (SX) superalloys are important high-temperature materials used for manufacturing turbine blades in aero-engines. During service under combinational impacts of temperature and stress, the SX superalloy may reach its life due to plastic deformation, which normally accompanies time-dependent microstructural degradation. To reveal this dynamically mechanical response, tensile tests at 1050°C are carried out to record stress-strain curves at five strain rates as well as creep curves at four applied stresses. Deformed microstructures and defects have been analyzed to understand mechanical behaviors and the underlying mechanism by using advanced scanning electron and scanning transmission electron microscopes. Results show that the deformation mode of the alloy strongly depends on the strain rates/applied stresses under mechanical loading. The dislocation density inside the γ phase is extremely low at all tests, indicating that the γ phase is relatively weak and ready to flow at this temperature even at a very fast strain rate. The deformation behavior of the γ′ phase is much complicated. At fast strain rates or high applied stresses, the dislocation density in the γ′ phase is very high, contributing to high-stress requirements to deform the material. At slow strain rates or low applied stresses, rafting microstructures develop and the deformation mode becomes directional coarsening/diffusion-dominated. Our results demonstrate a comprehensive understanding of the deformation mechanism of Ni-based SX superalloys, which may provide lifetime prediction of the mechanical failure, as well as the database for superalloy applications in mechanical systems.

Keywords
creep, defects, deformation behavior, microstructure, Ni-based single crystal superalloy, strain rate sensitivity
1 | INTRODUCTION

Ni-based single crystal (SX) superalloys are currently the most important material, which can be selected to manufacture turbine blades for aero-engines, due to their superior overall performance, including excellent high-temperature mechanical properties and good oxidation resistance.\textsuperscript{1,2} Composition and microstructure are the two key factors determining the performance of such alloy.\textsuperscript{1,2-7} Ni-based SX alloy generally consists of two coherent phases.\textsuperscript{8-11} One is the face-centered cubic (FCC) γ matrix phase, the other is the cubic γ′ strengthening phase with ordered L1₂ structure whose ideal size is about 300–500 nm. The γ′ precipitates are homogeneously distributed in the continuous γ matrix, with γ/γ′ interface along [001] planes after proper fabrication and heat treatment processing. The typical morphology of the two phases keeps changing when the alloy is under service conditions.\textsuperscript{12-15} For example, γ′ rafting, secondary γ′ precipitation, formation of interfacial dislocation network, and so forth, would occur when Ni-based SX alloys are exposed to high temperature, stress, and prolonged time, which eventually lead to material failure.\textsuperscript{16-17} Therefore, revealing deformation mechanism of an SX superalloy by understanding the relationship between microstructural evaluation and mechanical behavior under stress/temperature combination is the key research topic for SX superalloys. Moreover, at elevated temperature, microstructure degradation and material failure are time-related dynamic processes, and understanding how stress/temperature/time in combination affects the material degradation/failure is essential for part design, life prediction, and processing optimization of the alloy.

Ding et al.\textsuperscript{20,25} have systematically studied tensile properties of a second-generation SX alloy at 10⁻³ s⁻¹ strain rate and revealed mechanical responses and deformation mechanisms of the two phases at different temperatures from room temperature (RT) to 1100°C. Clearly, the geometry of both γ/γ′ phases varies throughout the entire deformation process, especially at high temperatures. For example, at a temperature of 1100°C, the strength of the γ matrix is very low and ready to flow under mechanical loading. Although a raft-like microstructure is observed at a normal strain rate (10⁻³ s⁻¹), but the mechanism seems to be different from that caused by the creep experiment. In a common tensile strain rate of 10⁻³ s⁻¹, the γ′ cubes are clearly elongated along the tensile direction (this is easy to imagine). The raft-like microstructure is caused mainly by Poisson’s effects where the γ phase in vertical channels is squeezed into horizontal channels making γ′ precipitates joining together. In traditional creep testing, however, initial γ′ cuboidal phases transform into plates or lamellar, which is also referred to as directional coarsening.\textsuperscript{21,22} For typical second-generation SX superalloys with negative lattice mismatch, so-called Type N (normal) rafting microstructures form where the cuboids coarsen preferentially transverse to the direction of the external tensile stress. For example, for a [001] orientated SX, if tensile loading is also along [001] direction, γ′ cuboidal particle will directionally coarsen in the (001) plane normal to the tensile direction; moreover, the thickness of the γ′ plates becomes shorter than the edge length of the γ′ cube. It is believed that the rafting microstructure during creep tests is caused by the directional diffusion of alloying elements.\textsuperscript{23,24}

The microstructure and mechanism comparison between normal tensile and traditional creep tests suggest that the deformation rate plays an important role in the mechanical behavior of SX Ni-based superalloys at elevated temperatures because diffusion is a time-dependent process. A comprehensive understanding of the rate-dependent mechanism, especially the dependence of stress and strain rate on the deformation behavior of the alloy, can provide lifetime prediction and the database for mechanical systems design for superalloy applications. Therefore, we design tensile tests at five strain rates and four applied stresses to obtain strain-rate-sensitive stress-strain curves and stress-dependent creep curves to reveal the rate-dependent mechanical behavior of a Ni-based SX superalloy at 1050°C. By using advanced scanning electron microscopy (SEM) and transmission electron microscopy (TEM), the microstructures and their evolution under the combinational effect of stress and strain rate are investigated to explore the rate-dependent deformation mechanism of Ni-based SX superalloys.

2 | MATERIALS AND METHODS

2.1 | Materials

The nominal composition of the selected Ni-based SX superalloy in this study is 4.2 Cr–8.8 Co–2.2 Mo–9.0 Ta–2.3 Re–0.5 Nb–5.1 Al–0.1 Hf–Ni (all in wt%). The fabrication processes of the SX superalloy are the same as those previously reported.\textsuperscript{20,25} Briefly, the SX superalloy is grown by using the Bridgman method, and homogeneous γ/γ′ two-phase microstructure is obtained after solid solution treatment (1290°C/1 h + 1300°C/2 h + 1315°C/2 h + 1325°C/4 h + 1330°C/4 h/air cooling [AC]) and subsequent two-step aging treatment (1120°C/4 h/AC + 870°C/24 h/AC). Figure 1 shows the typical homogeneous γ/γ′ two-phase microstructure in the SX superalloy. Cubic γ′ precipitates, with a size of about 400 nm, and distributes in the continuous γ matrix channel (Figure 1A). An annular dark-field scanning TEM (ADF-STEM) image (Figure 1B) shows that there are no dislocations, stacking faults, and other defects existing in the two phases and on the γ/γ′ interface. And the selected area diffraction patterns of the two phases (Figures 1C,D) indicate that the γ and γ′ phases are FCC and L1₂ structures, and the two phases are coherent.

2.2 | Mechanical testing

Dog bone-shaped specimens for tensile and creep tests, whose gauge section is 3.1 × 1.0 × 12.5 mm³, were machined from SX rods with typical microstructures shown in Figure 1. The tensile/creep direction of the specimens is along [001], and the other two side surfaces of the tensile/creep specimens are along (100) and (010), respectively. To remove microcracks introduced by electro-discharge machining, all the surfaces of the gauge section were carefully ground down with 800 grit SiC paper.
Tensile and creep tests were all conducted in air at 1050°C on a screw-driven mechanical testing machine equipped with an induction heater. During tensile/creep tests, a thermocouple, which was attached to the sample gauge section directly, was used to feedback-control and monitor the temperature of the samples. The testing temperature was within ±3°C of the set temperature. Tensile tests were conducted at engineering strain rates of $10^{-1}$, $10^{-2}$, $10^{-3}$, $10^{-4}$, and $2 \times 10^{-5}$ s$^{-1}$. Except for the test at $10^{-1}$ s$^{-1}$, all other tensile tests were interrupted when the strain has reached approximately 19%. Creep tests were conducted at applied tensile stresses of 500, 375, 325, and 250 MPa. Except for the test with applied stress at 250 MPa, where the test was interrupted at creep strain of ~10%, the other three creep experiments were all interrupted when creep strain reached approximately 15%.

### 2.3 Microstructure characterization

Standard metallographic procedures were used for preparing the SEM specimens, including grinding, mechanical polishing, and electro-etching. The specimens were ground to 1200 grit SiC paper first, then polished in an automatic vibrational polishing machine. The polished samples were electro-etched in pure phosphoric acid at RT to slightly remove the matrix, which is beneficial to SEM observation. SEM images were captured with viewing direction along [100] direction by using an FEI quanta 650 SEM with an accelerating voltage of 15 kV and a 10 mm working distance.

Disks with a diameter of 3 mm were machined from the deformed specimens along (100) planes. These disks were ground down to ~50 μm in thickness, then twin jet electrolytically in an alcoholic solution containing 5 vol% perchloric acid at ~30°C so as to obtain an electron transparent area used for TEM observation. A spherical aberration-corrected FEI Titan G2 scanning TEM, operated at 200 kV, was employed for TEM characterization, equipped with an ADF detector and a high-angle annular dark-field (HAADF) detector.

Figure 2A shows 1050°C engineering stress-strain curves of the SX superalloy at different strain rates ($10^{-1}$, $10^{-2}$, $10^{-3}$, $10^{-4}$, and $2 \times 10^{-5}$ s$^{-1}$). It is obvious that strain rate has a significant effect on stress-strain response of the alloy at this temperature, although all curves exhibit a similar trend. In all five strain rates, the flow stresses increase until the strain reaches approximately 1.5%, then decrease continuously until sample fracture or experiment interruption. The strength of the alloy increases significantly, where the stress-strain curves shift to higher stresses as the strain rate increases from $2 \times 10^{-5}$ to $10^{-1}$ s$^{-1}$. To quantitatively analyze the strain rate dependence, the flow stresses at a strain of 0.2% (yield), 2%, and 15% have been calculated and listed in Table 1. It is obvious that the higher the strain rate, the higher the strength. To visualize the strength versus strain rate relationships, data in Table 1 are plotted in Figure 2B. Clearly in log-log plot, straight lines can be used to well represent the power law relation between flow stress ($\sigma$) and strain rate ($\dot{\varepsilon}$) at a certain strain level,

$$\sigma = C_1 \dot{\varepsilon}^m,$$

where $C_1$ is a constant and $m_1$ is the strain rate sensitivity, which is the slope of the green, red, and blue dash lines in Figure 2B. Therefore, the strain sensitivity coefficients of the SX superalloy are 0.105 (0.2% strain), 0.093 (2% strain), and 0.101 (15% strain) at 1050°C. The stress sensitivity with $m_1 = -0.1$ is in the range of metallic alloys at elevated temperatures.

To reveal the microstructure changes after deformation in the otherwise homogeneous $\gamma'/\gamma$ two-phase SX superalloy, SEM images have been captured from uniformly deformed gauge sections after tensile testing and the results are shown in Figures 3A–E. When deformation is relatively quick with a high strain rate, the tensile test was finished in a very short time (~3 s for $10^{-1}$ s$^{-1}$), as shown in
The slopes of green, red, and blue dash lines are 0.105, 0.093, and 0.101, which respectively correspond to the strain sensitivity coefficient ($m_1$) at three different strain levels.

| Strain rate (s⁻¹) | Strength at 0.2% strain (MPa) | Strength at 2% strain (MPa) | Strength at 10% strain (MPa) |
|------------------|-------------------------------|----------------------------|-----------------------------|
| $10^{-1}$        | 848                           | 882                        | 816                         |
| $10^{-2}$        | 635                           | 691                        | 616                         |
| $10^{-3}$        | 490                           | 548                        | 493                         |
| $10^{-4}$        | 394                           | 439                        | 404                         |
| $2 \times 10^{-5}$ | 349                          | 404                        | 352                         |
| Strain sensitivity coefficient | 0.105                       | 0.093                      | 0.101                       |

Abbreviation: SX, single crystal.

Figure 3A, the $\gamma'$ precipitates are still separated but stretched along the tensile direction to form square prisms with spherical corners. Meanwhile, the horizontal matrix channel under tensile stresses is widened and the vertical channel (under compression due to Poisson’s effect) is narrowed. Microstructure of the sample deformed at $10^{-2}$ s⁻¹ (Figure 3B), whose deformation process to reach ~20% strain takes about 20 s, is similar to that deformed at $10^{-1}$ s⁻¹, except that the corners of the square prisms become rounder. When the strain rate further decreases to $10^{-3}$ s⁻¹, tensile testing takes about 200 s, as shown in Figure 3C. The degree of spheroidization becomes more severe in the corners of $\gamma'$ precipitates, in the meanwhile, vertical $\gamma$ channels seem to be narrower, and horizontal $\gamma$ channels seem to be wider than those deformed at much higher strain rates (Figures 3A,B). In addition, some precipitates start to coalesce from the corner to form bands, as indicated by red arrows in Figure 3C, leaving vertical lath-shaped $\gamma$ phase between $\gamma'$ bands. Note that many $\gamma'$ precipitates are still not connected. When the strain rate is further decreased to $10^{-4}$ s⁻¹ (tensile process takes ~35 min), as shown in Figure 3D, $\gamma'$ precipitates coalesce from the corners and form rafting-like microstructure, and the majority of residual $\gamma$ phases in the rafted $\gamma'$ band are lenticule-like (indicated by blue arrows in Figure 3D) or spherical shapes (indicated by white arrows in Figure 3D). When tensile time prolongs to ~200 min at a strain rate of $2 \times 10^{-5}$ s⁻¹, microstructure (Figure 3E) is similar to that deformed at $10^{-4}$ s⁻¹, but the rafted $\gamma'$ bands seem to be more continuous, and the residual $\gamma$ phases in $\gamma'$ are all spherical, indicating that $\gamma'$ precipitates almost coalesce together to form continuous bands.

The effects of temperature (thermal coarsening) and stress (deformation) on the microstructure are quantified as size variations of both phases at high temperatures with and without stress. Microstructure in the grip area of tensile tested specimens, as shown in Figure S1, see Supporting Information, represents locations where the changes of phases are solely due to thermal effects. The microstructure in the gauge section (e.g., Figures 3A,B) represents the locations where changes are due to both thermal and stress effects. Therefore, we measure the lengths of both phases along the tensile direction in both grip and gauge sections and the result is shown in Figure 3F. The upper part of Figure 3F is the percent of length changes of both $\gamma$ and $\gamma'$ phases compared to the initial phase sizes (e.g., Figure 1) in the grip section (without deformation, Figure S1, see Supporting Information), reflecting thermal coarsening effects. The bottom part of Figure 3F is the percent changes of phase sizes between gauge and grip area (e.g., Figures 3 and S1, see Supporting Information), reflecting the stress effects. Clearly as shown in the upper part of Figure 3F, both phases at the grip area coarsen more in the lower strain rate. When the strain rate is lower than $2 \times 10^{-5}$ s⁻¹ (test time is ~200 min), size variations of the two phases reach 31% ($\gamma'$ phase) and 16.8% ($\gamma$ phase), which also indicate that $\gamma'$ precipitates are redissolved into the $\gamma$ phase except for the coarsening of the two phases. As shown in the bottom part of
Figure 3F, it is found that both $\gamma$ and $\gamma'$ phases at the gauge section are longer than those at the grip area, indicating partly tensile force effects. Length changes of $\gamma$ phase are all higher than 46%, indicating that plastic strains accumulated in $\gamma$ matrix are more significant than that accumulated in $\gamma'$ phase, as the total plastic strain of the alloy is less than 20%. Another reason for the big length changes of $\gamma$ channels normal to tensile direction is that the materials in vertical $\gamma$ channels are squeezed to the horizontal channels due to Poisson’s effect, both suggesting that the $\gamma$ phase is much weaker than $\gamma'$ phase at this temperature. In addition, with decreased strain rate and prolonged deformation time, the size variation of $\gamma$ channels perpendicular to tensile direction monotonically increases from 46% (strain rate $10^{-1}$ s$^{-1}$) to 107% (strain rate $2 \times 10^{-5}$ s$^{-1}$), demonstrating that deformation of $\gamma$ becomes increasingly severe with decreased strain rate. In contrast, the size variation of $\gamma'$ precipitates decreases with decreased strain rate. For example, it is 27.9% at strain rate of $10^{-1}$ s$^{-1}$, and decreases to 11% for strain rate of $10^{-4}$ s$^{-1}$ and 13.6% for strain rate of $2 \times 10^{-5}$ s$^{-1}$. This may be caused by directional coarsening/diffusion, which compensates for the stress-induced elongation, similar to the rafting in creep test, which will be presented in the following sections.

3.2 The applied stress effects

Figure 4A shows creep curves of the SX superalloy, illustrating the relationship between creep strain and time under four different applied stresses. It is obvious that the evolution of creep strain is remarkably sensitive to the applied stress. The lower the stress, the longer the time for the sample to reach a certain strain. Due to the huge time difference for the four creep curves in Figure 4A, the detail of the creep curve at 500 MPa has been shown in Figure 4B. The specimen crept at 500 MPa reached 10% strain in 6 min, while the
specimen crept at 250 MPa needed more than 15 h to reach 10% strain. Despite the huge difference in creep time, all curves exhibit similar trends, which contain general three stages of creep. Take the curve at 250 MPa for example. The creep strain accumulations drop first and reach the minimum at about 1% strain (known as primary creep). Then, the creep strain is approximately constant and referred to as secondary creep. Subsequently, it witnesses a stage in which creep strain increases with increasing strain until interruption or specimen rupture, which is called tertiary creep. For further analysis of creep behaviors of the alloy, creep rates at different strains have been calculated and plotted in Figure 4C in which variations of creep rates at three stages are clear. The minimum creep rate ($\dot{\epsilon}_{\text{min}}$) can be obtained from Figure 4C and summarized in Figure 4D. $\dot{\epsilon}_{\text{min}}$ is an important reference index for design purposes, and is related to the applied stress ($\sigma_{\text{app}}$) by the relationship:\ref{eq:creep_rate}

\begin{equation}
\dot{\epsilon}_{\text{min}} = C_2 \sigma_{\text{app}}^{m_2}
\end{equation}

where $C_2$ is a constant and $m_2$ is a stress exponent. All data can be represented by a Norton law (the red dash line in Figure 4D) with a stress exponent $m_2 = 8.6$ and Equation (2) can be written as $\dot{\epsilon}_{\text{min}} = 1.12e^{-27}\sigma_{\text{app}}^{8.6}$. This equation can be used for life prediction to reach certain strain and safe stress prediction of this SX alloy under 1050°C service.

The 1050°C crept microstructure has also been investigated to reveal the creep mechanisms and results are shown in Figure 5. When the stress is relatively high (500 MPa), creep strain reaches 15% in 8 min. The $\gamma'$ precipitates are stretched along the tensile direction and most $\gamma'$ have coalesced from the corners, with narrowed lathy vertical $\gamma$ channel and widened horizontal $\gamma$ channel (Figure 5A). When stress decreases to lower than 375 MPa, raft-like $\gamma'$ precipitates are formed (Figures 5B–D), but the size and density of the residual vertical $\gamma$ phase in rafted $\gamma'$ decrease with decreased stress. For example, there are still many residual spherical $\gamma$ phases in $\gamma'$ of the specimen crept at 375 MPa (indicated by red arrows in Figure 5B), while residual $\gamma$ phase is not observed in $\gamma'$ phase in the specimen crept at 250 MPa (Figure 5D).

To quantitatively analyze the influence of stress on the deformation microstructure of the alloy, the sizes of the two phases in the gauge section and grip area along the tensile direction are
measured using the same method as described in Figure 3F after creep tests. Figure 5E is the percentage length changes of both γ and γ' phases compared to the initial phase sizes in the grip section (without deformation, Figure S2, see Supporting Information), reflecting thermal coarsening effects, the same as Figure 3F, upper part. Figure 5F shows the phase length changes of γ and γ' phases between the gauge section and the grip area, which is also similar to that observed in Figure 3F, bottom part. As deformation time prolongs in creep tests, for example, at the stress of 250 MPa, the γ phase gets wider and the size variation reaches 142% where creep time is up to 15 h with creep strain of ~10%. Interestingly, size variations of γ' phase in creep test are all lower than 10% and even become a negative number when creep stress is at 250 MPa, making the length of γ' phase even short in the tensile direction. The possible reasons will be discussed next based on directional diffusion/coarsening in creep of Ni-based SX superalloys.

**Figure 5**  Microstructure of the single crystal superalloy after creep testing at 1050°C with four different applied stresses. (A) 500 MPa, (B) 375 MPa, (C) 325 MPa, (D) 250 MPa. As applied stresses decrease from 500 MPa (A) to 250 MPa (D), microstructures indicating the deformation changes from stress dominated to directionally coarsening dominated (rafting). Red arrows in (B) indicate the residual γ phase in the rafted γ'. (E,F) The size changes of γ and γ' phases after creep testing as a function of applied stresses. Sizes of phases are defined as the length of γ' and thickness of γ phases along the tensile direction. (E) Size changes at grip section (without deformation) reflecting thermal coarsening of both phases. (F) The percentage length changes between gauge and grip area, reflecting the stress effects. Note that every data point in (E,F) is based on more than 50 measurements in both phases. Dash line in (E,F) indicates that size variation is zero.

### DISCUSSION

Plastic deformation of alloys accompanies by the motion of defects, including vacancies, dislocations, stacking faults, and so forth. The changes of geometry and sizes of the two phases no matter in tensile (Figure 3) or creep (Figure 5) at 1050°C are related to defect generation and material flow behaviors. TEM images, for samples after both tensile tests with different strain rates (Figure 6) and creeps with different applied stresses (Figure 7), are captured using ADF-STEM imaging mode to investigate the possible deformation behavior and mechanism.

Figure 6 shows TEM images of specimens tensile deformed at different strain rates. There is almost no dislocation existing in the γ matrix. This may result from high dislocation mobility and relatively weak strength of this phase at 1050°C, which is consistent with the higher size variation of γ phase at gauge section than that of γ' phase.
FIGURE 6  Annular dark-field scanning transmission electron microscopy images of the single crystal superalloy after 1050°C tensile testing with different strain rates. (A) $10^{-1}$ s$^{-1}$. Large amounts of dislocations existing in the $\gamma'$ precipitates. (B) $10^{-3}$ s$^{-1}$. The dislocation density in $\gamma'$ is much lower. (C) $10^{-4}$ s$^{-1}$. Dislocations are mainly on the interface, and no dislocation inside the $\gamma'$ phase. (D) $2 \times 10^{-5}$ s$^{-1}$. SX alloys exhibit rafting microstructure, with small amounts of residual $\gamma$ phases remaining inside $\gamma'$ bands (red arrows). Dislocations are lying on the interface. Note that in all samples tensile tested at four strain rates, $\gamma$ phase is almost dislocation-free after deformation.

FIGURE 7  Annular dark-field scanning transmission electron microscopy images of the single crystal superalloy after creep tests at 1050°C with four different applied stresses. (A) 500 MPa, (B) 375 MPa, (C) 325 MPa, (D) 250 MPa. (A) High-density dislocations in the $\gamma'$ precipitates of sample crept at 500 MPa. (B) At applied stress of 375 MPa, there are still dislocations and residual $\gamma$ phase existing in the rafted $\gamma'$ bands. (C) At creep stress of 325 MPa, rafting microstructures is similar to (B) with less dislocation and residual $\gamma$ phases. (D) At a stress of 250 MPa, rafting is more obvious, and dislocations are only lying on the interface and no dislocation inside the $\gamma'$ bands. Note that in all four applied stresses, the $\gamma$ phase is almost dislocation-free after creep tests.
The γ' phase behaves very differently as strain rates change. When the strain rate is high and deformation is fast (e.g., at $10^{-1}$ s$^{-1}$, deformation takes only ~3 s), there are large amounts of dislocations existing in γ' precipitates (Figure 6A). When strain rate decreases to $10^{-3}$ s$^{-1}$, dislocation density in γ' (Figure 6B) is much lower than that in γ' tensile deformed at $10^{-3}$ s$^{-1}$. When deformed at $10^{-4}$ s$^{-1}$, γ' precipitates are almost dislocation-free, and dislocations are observed mainly on the γ/γ' interface (Figure 6C). At this strain rate, γ' precipitates seem to be rafting in SEM image (Figure 3D), but they are still separated by interfacial dislocation network in TEM (Figure 6C). With strain rate further decreased to $2 \times 10^{-5}$ s$^{-1}$, as shown in Figure 6D, the γ' actually are rafted with almost no dislocation inside the entire γ' bands, but containing little spherical residual γ-phase, which is consistent with SEM observation (Figure 3E). The high dislocation density in the γ' phase at a high strain rate suggests that those dislocations do not have time to escape from the γ' phase, contributing to the high-stress requirement to deform the material. Meanwhile, the γ phase is relatively weaker and ready to flow, thus the γ phase in the vertical channels is squeezed into the horizontal channels, which is consistent with previous studies.20 When strain rates decrease and the deformation processes prolong, time becomes enough to allow dislocation escape and deposit into the interfaces, and the strength decreases. Moreover, directional diffusion/coarsening become more dominated, which can be clearly observed in the creep specimens.

Figure 7 is ADF-STEM images of the SX superalloy after creep tests at 1050°C with four different applied stresses. There is a large amount of dislocations in the γ' phase of the sample crept at 500 MPa, as shown in Figure 7A, suggesting the similarity between creep at high applied stress and tensile at high strain rate. In both cases, strengths are relatively high and deformation processes are very fast, resulting in a high density of dislocations in γ', which do not have enough time to escape from inside to the interfaces. When creep stresses decrease to lower than 375 MPa and deformation time prolongs to more than 1.8 h, dislocation densities in γ' phase are low and most dislocations are lying on the γ/γ' interface (Figures 7B-D). When creep stress decreases to 250 MPa, it takes more than 15 h to reach 10% plastic stain and γ' rafts develop, where tensile stress in horizontal channels might lead to higher vacancy concentration and therefore higher diffusion rates of constituent elements. In addition, with prolonged time at high temperature, γ' forming elements may redissolve back to γ phase from the horizontal interface, which is normal to tensile direction, leading to a phenomenon that the γ' at the tensile direction becomes even short (opposed to otherwise elongation along the tensile direction). Clearly, at low applied stress or low strain rate, the deforming mode mainly is a creep, which is controlled by elemental diffusion and redissolution, resulting in directionally coarsening (rafting) microstructure.14,16,30

Collectively, the effects of stress rate and applied stress on deformation behavior and mechanism of the SX superalloy at 1050°C are summarized in Figure 8. Strain rate versus strength or creep rate versus applied stress curves all follow classic power law relationships described in Equations (1) and (2) (schematically represented as a red line in log-log plot of Figure 8). The deformation mode of the alloy strongly depends on the strain rates/applied stresses under mechanical loading. The γ phase at 1050°C is relatively weak and ready to flow at this temperature in all current tests. The dislocation density inside the γ phase is extremely low even in the fastest deformation. The materials in vertical channels (under compression) are squeezed out into horizontal channels (under tensile). Besides the direct effects of stress, tensile stress in horizontal channels would lead to a higher density of vacancies, which prompts the element’s directional diffusing from vertical channels to horizontal channels. The latter case is more pronounced at a low strain rate or low creep stress because the deformation time is prolonged.

The deformation behavior of the γ' phase is much complicated. At high strain rates or high applied stresses, the dislocation density in the γ' phase is very high, indicating a high-stress requirement to deform the material. In this case, plastic deformation stretches the γ' phase along the tensile direction, and we call this stage “stress-dominated.” At low strain rates or low applied stresses, rafting microstructures develop and the deformation mode becomes directional coarsening/diffusion-dominated. The γ' phase near upper and lower interface would directionally redissolve into the horizontal γ channels, leading to shorter γ' precipitates along the tensile direction. In this case traditional rafting,5,16,18,31 as observed in creep testing are the main deformation mode. It appears that although both phases accumulate plastic strain during deformation, the matrix contributes more strain no matter through diffusion or flow. Therefore, the alloy design strategy for Ni-based superalloy should focus more on the matrix. When the application requires high strength and serves at a short time, alloy design should focus on strengthening of γ phase. Meanwhile, if service conditions require a long lifetime at high temperatures, alloy design should focus on decreasing elemental diffusion in the γ phase.

**FIGURE 8** Deformation map schematically illustrating the effect of applied stress and strain rate on the deformation behavior and mechanism of a Ni-based single crystal superalloy.
5 | SUMMARY AND CONCLUSIONS

To understand how stress/temperature/time in combination affects the material degradation/failure, tensile tests at 1050°C are carried out at five different strain rates and four applied stresses to obtain stress-strain and creep curves. With the help of advanced SEM and TEM technologies, the evolution of deformation substructures and defects are investigated to reveal the underlying mechanisms. Both strain rate ($\dot{\varepsilon}$) versus strength ($\sigma$) and creep rate ($\dot{\varepsilon}_{\text{min}}$) versus applied stress ($\sigma_{\text{app}}$) curves follow classic power law relationships. For strain rate sensitivity, $\sigma = C_1 \dot{\varepsilon}^{0.10}$; for stress dependence creep, the minimum creep rate $\dot{\varepsilon}_{\text{min}} = 1.12 \varepsilon^{2.7} \sigma^{0.6}$.

The deformation mode of the alloy strongly depends on the strain rates/applied stresses under mechanical loading; the γ phase is relatively weak and ready to flow at 1050°C even at very fast deformation mode. The deformation behavior of the γ′ phase is much complicated as a function of strain/stresses. At high strain rates or high applied stresses, the dislocation density in the γ′ phase is very high, which contributes to the high-stress requirement to deform the material where deformation mode is stress/dislocation-dominated. At low strain rates or low applied stresses, plastic deformation results in rafting microstructures due to directional coarsening where the deformation mode becomes diffusion-dominated.

ACKNOWLEDGMENTS

This study was supported by the Basic Science Center Program for Multiphase Media Evolution in Hypergravity of the National Natural Science Foundation of China (No. 51988101), the Innovation Fund of the Zhejiang Kechuang New Materials Research Institute (No. ZKN-20-P01, ZKN-20-201), the Fundamental Research Funds for the Central Universities (No. 2020QNA4004), the Key R & D Project of Zhejiang Province (No. 2020C01002), Natural Science Foundation of Zhejiang Province (No. LQ20E01008), and National Natural Science Foundation of China (No. 91960201).

CONFLICT OF INTEREST

The authors declare that there are no conflict of interest.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

ORCID

Hongbin Bei http://orcid.org/0000-0003-0283-7990

REFERENCES

1. Reed RC. The Superalloys: Fundamentals and Applications. Cambridge University Press; 2006.
2. Pollock TM. Alloy design for aircraft engines. Nat Mater. 2016;15: 809-815.
3. Zhang J, Li J, Jin T, Sun X, Hu Z. Effect of Mo concentration on creep properties of a single crystal nickel-base superalloy. Mater Sci Eng A. 2010;527:3051-3056.
4. Murakumo T, Kobayashi T, Koizumi Y, Harada H. Creep behaviour of Ni-base single-crystal superalloys with various γ′ volume fraction. Acta Mater. 2004;52:3737-3744.
5. Zhang JX, Murakumo T, Harada H, Koizumi Y, Kobayashi T. Creep deformation mechanisms in some modern single-crystal superalloys. Superalloys. 2004:189-195.
6. Reed RC, Tao T, Warnken N. Alloys-by-design: application to nickel-based single crystal superalloys. Acta Mater. 2009;57:5898-5913.
7. Yokokawa T, Harada H, Mori Y, et al. Design of next generation Ni-base single crystal superalloys containing Ir: towards 1150°C temperature capability. Superalloys. 2016;123-130.
8. Ding Q, Li S, Chen LQ, et al. Re segregation at interfacial dislocation network in a nickel-based superalloy. Acta Mater. 2018;154:137-146.
9. Collins DM, Yan L, Marquis EA, et al. Lattice misfit during ageing of a polycrystalline nickel-base superalloy. Acta Mater. 2013;61:7791-7804.
10. Nabarro FRN. Rafting in superalloys. Metall Mater Trans A. 1995;27: 513-530.
11. Ding Q, Lao Z, Wei H, Li J, Bei H, Zhang Z. Site occupancy of alloying elements in γ′ phase of nickel-base single crystal superalloys. Intermetallics. 2020;121:106772.
12. Nazmy M, Epishin A, Link T, Staubli M. A review of degradation in single crystal nickel based superalloys. Energy Mater. 2006;1:263-268.
13. Graverend JBL, Cormier J, Jouiad M, Gallerneau F, Paulmier P, Hamon F. Effect of fine γ′ precipitation on non-isothermal creep and creep-fatigue behaviour of nickel base superalloy MC2. Mater Sci Eng A. 2010;527:5295-5302.
14. Reed RC, Matan N, Cox DC, Rist MA, Rae CMF. Creep of CMSX-4 superalloy single crystals: effects of rafting at high temperature. Acta Mater. 1999;47:3367-3381.
15. Epishin A, Link T, Nazmy M, Staubli M, Klingelhofer H, Nelson J. Microstructural degradation of CMSX-4: kinetics and effect on mechanical properties. Superalloys. 2008:725-731.
16. Reed RC, Cox DC, Rae CMF. Damage accumulation during creep deformation of a single crystal superalloy at 1150°C. Mater Sci Eng A. 2007;448:88-96.
17. Moverare JJ, Johansson S, Reed RC. Deformation and damage mechanisms during thermal-mechanical fatigue of a single-crystal superalloy. Acta Mater. 2009;57:2266-2276.
18. Tian S, Su Y, Qian B, Yu X, Liang F, Li A. Creep behavior of a single crystal nickel-based superalloy containing 4.2% Re. Mater Des. 2012; 37:236-242.
19. Yuan Y, Kawagishi K, Koizumi Y, Kobayashi T, Yokokawa T, Harada H. Creep deformation of a 6th generation Ni-base single crystal superalloy at 800°C and 735 MPa. Superalloys. 2016:675-682.
20. Ding Q, Bei H, Yao X, et al. Temperature effects on deformation substructures and mechanisms of a Ni-based single crystal superalloy. Appl Mater Today. 2021;23:101601.
21. Jácome LA, Nörtershäuser P, Heyer JK, et al. High-temperature and low-stress creep anisotropy of single-crystal superalloys. Acta Mater. 2013;61:2926-2943.
22. Carroll LJ, Fong Q, Pollock TM. Interfacial dislocation networks and creep in directional coarsened Ru-containing nickel-base single-crystal superalloys. Metall Mater Trans A. 2008;39:1290-1307.
23. Pollock TM, Argon AS. Directional coarsening in nickel-base single crystals with high volume fractions of coherent precipitates. Acta Metall Mater. 1994;42:1859-1874.
24. Reed RC, Cox DC, Rae CMF. Kinetics of rafting in a single crystal superalloy: effects of residual microsegregation. Mater Sci Technol. 2007;23:893-902.
25. Ding Q, Bei H, Zhao X, Gao YF, Zhang Z. Processing, microstructures and mechanical properties of a Ni-based single crystal superalloy. Crystals. 2020:10:572.
26. Ding Q, Bei H, Wei X, Gao YF, Zhang Z. Nano-twin-induced exceptionally superior cryogenic mechanical properties of a Ni-based GH3536 (Hastelloy X) superalloy. Mater Today Nano. 2021;14:100110.
SUPPORTING INFORMATION

Additional supporting information may be found in the online version of the article at the publisher’s website.

How to cite this article: Ding Q, Bei H, Li L, et al. The dependence of stress and strain rate on the deformation behavior of a Ni-based single crystal superalloy at 1050°C. Int J Mech Syst Dyn. 2021;1:121-131.
doi:10.1002/msd2.12002