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# EBSD analysis of evolution of dynamic recrystallization grains and δ phase in a nickel-based superalloy during hot compressive deformation

Y.C. Lin [a,b,c,*], Dao-Guang He [a,b], Ming-Song Chen [a,c], Xiao-Min Chen [a,c], Chun-Yang Zhao [a,b], Xiang Ma [d], Zhi-Li Long [e]

[a] School of Mechanical and Electrical Engineering, Central South University, Changsha 410083, China
[b] Light Alloy Research Institute of Central South University, Changsha 410083, China
[c] State Key Laboratory of High Performance Complex Manufacturing, Changsha 410083, China
[d] SINTEF Materials and Chemistry, P.O.Box 124, Blindern, 0314 Oslo, Norway
[e] Harbin Institute of Technology, Shenzhen Graduate School, Shenzhen 518055, China

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ABSTRACT

Hot compression tests of a nickel-based superalloy with δ phase ($Ni_3Nb$) are performed. The evolution of dynamic recrystallization (DRX) grains and δ phase, as well as the interactions between DRX grains and δ phase, is investigated. It is observed that the fraction of high-angle grain boundaries significantly increases with the increased true strain or the decreased strain rate. This attributes to the obvious DRX nucleation at relatively low strain rates or large true strains. The fraction of high-angle grain boundaries firstly increases, and then decreases when the temperature is increased. It is related to the fact that δ phases sharply dissolute as the deformation temperature exceeds 980 °C, and the restricting effects of δ phases on the growth of DRX grains are significantly weaken. Meanwhile, the initial needle-like δ phases experience the distortion, dissolution and transformation into the rod-like δ phases during hot compression deformation. Besides, δ phase can easily induce high density dislocation cells, which accelerates the formation and rotation of subgrains. So, nucleation of DRX is stimulated by δ phase.



## 1. Introduction

Nickel-based superalloys are extensively used to manufacture the critical high-temperature components (turbine disks, engine casings, etc.) in aviation industries, because of the good mechanical properties and oxidation resistance at elevated temperatures [1–4]. Generally, most of high-temperature components are prepared by hot deformation. It is commonly known that the hot deformation behavior of nickel-based superalloys strongly depends on deformation parameters (strain, strain rate, deformation temperature, etc.) [5,6]. Meanwhile, several microstructural changes (dynamic recrystallization, phase transformation, etc.) occur during hot deformation, which in return affect deformation process. So, it is vital to investigate the flow behaviors and microstructural evolution of nickel-based superalloys during hot deformation.

Recently, the hot deformation behaviors and microstructural evolution of nickel-based superalloys have been intensively researched [7–35]. On the one hand, several suitable constitutive models, including phenomenological constitutive models [7–12] and physically-based constitutive model [13], were developed to predict the hot deformation behavior of some nickel-based superalloys. On the other hand, in order to optimize processing parameters, the processing maps of some nickel-based superalloys were developed by Wen et al. [14,15], Liu et al. [16], Zhang et al. [17], Wang et al. [18], Zhang et al. [19], Jiang et al. [20], and He et al. [21]. Besides, the microstructural changes of several nickel-based superalloys during hot deformation were investigated by Lin et al. [22,23], Chamanfar et al. [24], Lin et al. [25], Mitsche et al. [26], Somani et al. [27], Yeom et al. [28], Momeni et al. [29], Cheng et al. [30], Zhang et al. [31,32], Liu et al. [33], Reyes et al. [34], and Chen et al. [35]. Among the above researches, Electron Backscatter Diffraction (EBSD), Scanning Electron Microscope (SEM), Transmission Electron Microscopy (TEM), and Optical Microscope (OM) are main techniques to analyze the microstructural change during hot deformation. Especially, EBSD technique is increasingly used to comprehensively understand the change of grain structure and grain boundary misorientation distribution of the hot deformed metals or alloys [36–41].

Despite several investigations devoted to study the flow characteristic and microstructural evolution of some nickel-based superalloys, there are still limited literatures focusing on the evolution of dynamic recrystallization (DRX) grains and δ phase, as well as the interactions between DRX grains and δ phases. In present context, the influences

* Corresponding author at: School of Mechanical and Electrical Engineering, Central South University, Changsha 410083, China.
*E-mail addresses:* yclin@csu.edu.cn, linyongcheng@163.com (Y.C. Lin).

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of deformation parameters on the evolution of δ phase and DRX grains are investigated. Meanwhile, the interactions between DRX grains and δ phase are studied in detail.

## 2. Material and experimental

The used material was a commercial superalloy with the chemical compositions (wt%) of 52.82Ni–18.96Cr–5.23Nb–3.01Mo–1.00Ti–0.59Al–0.03C–0.01Co and (Bal) Fe. Cylindrical specimens (Φ 8 mm × 12 mm) were cut from the billet. The experimental specimens were firstly solution treated at 1045 °C for 45 min followed by cold water quenching. Then, all solution-treated specimens were aged at 900 °C for 12 h, and again quenched by cold water. Hot compression tests were performed on a Gleeble 3500 at 950–1010 °C and 0.001–0.1 $s^{-1}$. Before loading, all specimens were heated to the experimental temperatures at 10 °C/s, and then held for 5 min to eliminate the temperature gradient in specimens. The height reductions of specimens were selected from 30% to 70%. After hot compression tests, the tested specimens were immediately quenched by cold water. The microstructures of specimens were investigated by Electron Backscatter Diffraction (EBSD), Scanning Electron Microscope (SEM), Transmission Electron Microscopy (TEM), and Optical Microscope (OM). For OM and SEM observations, the specimens were mechanically polished, and chemically etched in a cupric chloride solution (50 ml HCl, 50 ml $CH_3CH_2OH$ and 2.5 g $CuCl_2$). For TEM and EBSD observations, the foils were machined from the compressed specimens, and grinded to be 70–80 μm thick, then the disks of 3 mm diameter were punched. Finally, the disks were electro-polished in a chemically corrosive solution (30 ml $HClO_4$ and 270 ml $CH_3CH_2OH$). The initial microstructure of the studied superalloy is illustrated in Fig. 1. It can be observed that the initial microstructure is mainly composed of needle-like δ phases, equiaxed grains and annealing twins. The content of initial δ phases can be statistically evaluated as 12.75%.

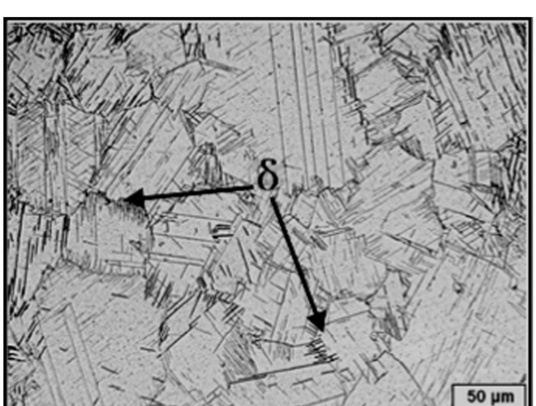


**Fig. 1.** Initial microstructure of the studied superalloy.

## 3. Results and discussion

### 3.1. High-temperature compressive deformation characteristics

#### 3.1.1. Typical true stress-true strain curve

Fig. 2 displays the typical true stress-true strain curves under tested deformation temperatures and strain rates. Evidently, the true stress abruptly increases to a peak value with increasing true strain, and then progressively decreases till a steady state. The variation of true stress with true strain is closely related to the competing process of work hardening and flow softening [25,42,43]. In the initial deformation stage, the dynamic recovery induced by dislocation cross-slip, climb and annihilation is weak because the stacking fault energy of the studied superalloy is low [1,29]. While the work hardening induced by dislocation multiplication and δ phase is obvious. Hence, the true stress rapidly increases to a peak value. As the true strain is further increased, the true stress gradually decreases. It results from the occurrence of dynamic recrystallization (DRX). During dynamic recrystallization, the nucleation and growth of new grains result in obvious dislocation annihilation, and then the work hardening is weaken [33,44,45,46]. With the further decrease of work hardening rate, the dynamic softening rate is balanced, and the steady stress appears. Besides, the true stress is distinctly affected by strain rate and deformation temperature. While the strain rate is decreased or the temperature is increased, the true stress decreases. This is because dislocation annihilation and grain boundary motion are accelerated as the deformation temperature is increased. Meanwhile, the time for the nucleation and growth of DRX grains is increased with decreasing strain rate [25,36,47].

#### 3.1.2. Determining critical strain ($\varepsilon_c$) for dynamic recrystallization

To investigate the microstructural changes in DRX process, the critical strain ($\varepsilon_c$) for initiating DRX should be firstly determined. Generally, $\varepsilon_c$ can be determined by $\theta-\sigma$ curves [13,48]. Here, $\theta=\frac{d\sigma}{d\varepsilon}$ and $\varepsilon$ are work hardening rate and strain, respectively. The detailed procedures are as follows. By the experimental true stress-strain curves, $\theta-\sigma$ curves can be plotted, and $\theta$ can be expressed as a third order polynomial function.

$$\theta = A_1\sigma^3 + A_2\sigma^2 + A_3\sigma + A_4 \tag{1}$$

where, $A_1$, $A_2$, $A_3$ and $A_4$ are material constants.

Taking the second derivative of Eq. (1) gives,

$$\frac{d^2\theta}{d\sigma^2} = 6A_1\sigma + 2A_2. \tag{2}$$

If $\frac{d^2\theta}{d\sigma^2}=0$, the critical stress ($\sigma_c$) can be determined as,

$$\sigma_c = -A_2/3A_1. \tag{3}$$

Then, $\varepsilon_c$ can be determined by the calculated $\sigma_c$ and true stress-strain curves. Fig. 3 shows the evaluated $\varepsilon_c$ under all tested conditions. From Fig. 3, it is observed that $\varepsilon_c$ increases with increasing strain rates or decreasing deformation temperatures. Furthermore, the values of $\varepsilon_c$ under all tested conditions are less than 0.2.

### 3.2. Influences of deformation parameters on the evolution of DRX grains

#### 3.2.1. Influences of true strain

To study the influences of true strain on microstructural evolution during dynamic recrystallization, the hot compression experiments with the final true strains of 0.36, 0.8 and 1.2 were carried out, respectively. Fig. 4 illustrates the influences of true strain on orientation imaging microscopy (OIM) maps and misorientation angle distribution at 950 °C and 0.01 $s^{-1}$. Obviously, the grains structures are drastically affected by true strain. At the true strain of 0.36, the serrated grains and bulging boundaries appears, as indicated in Fig. 4a. Meanwhile, there are some small DRX grains can be found along the original grain boundaries. It is commonly known that the serrated grain boundaries retain high strain gradient, and become the priority sites for the nucleation of DRX grains [25]. Moreover, the bulging of grain boundaries promotes the growth of DRX grains [25]. Besides, the DRX degree is small, which attributes to the low deformation storage energy at the true strain of 0.36. With increasing the true strain, the number of DRX grains sharply increases, as shown in Fig. 4c and e. This phenomenon results from the increased deformation storage energy when the true strain is increased. High deformation storage energy can bring the strong driven force for the movements of dislocation and grain boundary. Besides, it is found that part of DRX grains have grown up, when the true strain is further increased to 1.2, as illustrated in Fig. 4e. In addition, from Fig. 4b, d, and f, the average misorientation angles at the true strains of 0.36, 0.8