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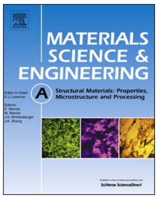

Behavior and modeling of microstructure evolution during metadynamic recrystallization of a Ni-based superalloy

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ABSTRACT

The metadynamic recrystallization (MDRX) behaviors of a Ni-based superalloy were studied by uniaxial compression tests with temperatures between 1223 and 1373 K and strain rates between 0.01 and 1 s^{-1}, followed by subsequent isothermal annealing for 0–60 s. Electron backscatter diffraction (EBSD) technique was employed to investigate the effect of annealing time, temperature and strain rate on the evolution of microstructure and twin boundaries during MDRX. The results showed that the recrystallized fraction and the number of high angle grain boundaries (HAGBs) increased with increasing annealing time and temperature. Increasing the strain rate can accelerate the MDRX process. Most low angle grain boundaries (LAGBs) were absorbed by migrating HAGBs, and a tiny fraction of LAGBs was transformed into HAGBs via subgrain growth. In addition, the $\sum 3^n$ boundaries first increased with increasing holding time and then decreased due to grain growth. Based on the experimental results, a dislocation density-based model to describe the microstructure evolution during MDRX was proposed and calibrated. Comparisons of the predicted and experimental recrystallized fraction indicated that the proposed model, which includes the relevant underlying microscale mechanisms, can accurately describe the MDRX behavior of the studied superalloy.

1. Introduction

Ni-based superalloys are being widely used in the aeronautic industry to manufacture critical parts which are employed in extreme in-service conditions of high stress and cyclic loading at elevated temperatures. The hot forming is commonly used to process these components. During hot forming, the material undergoes different cycles of heating, holding, deformation and cooling [1], which lead to the occurrence of grain growth, dynamic recrystallization (DRX), static recrystallization (SRX) and MDRX [2,3]. These physical processes significantly change the microstructure of the parts and then affect the mechanical properties of the final products. Generally, when the accumulated dislocation density exceeds the threshold value and triggers the DRX, MDRX will occur while the material is still at high temperature after deformation. Because no incubation period is required, the kinetics of MDRX is much more rapid than that of SRX which follows deformation in the absence of DRX [4]. The rapid MDRX kinetics then can produce a high softening rate and alter the microstructure significantly in a few seconds. Therefore, investigating and modeling the microstructure evolution during MDRX of Ni-based superalloys are important for optimizing the process parameters to meet the increasing demands for manufacturing high performance turbojet engines.

The flow behavior [4–8], microstructure evolution [5–7], and grain boundary character [8–11] during DRX of Ni-based superalloys have been extensively studied. However, the investigations on microstructure evolution, especially the grain orientation and twin boundary evolution during MDRX are relatively few. Park et al. studied the flow behavior and microstructure evolution of alloy 718 during DRX and MDRX by using single and two-step compression tests. It was found that the MDRX grain size depends on the Zener-Holloman (Z) parameter as well as on the initial grain size and applied strain [12]. Later, based on the Johnson–Mehl–Avrami–Kolmogorov (JMAK) models, Na et al. [13] developed a series of empirical equations to predict the average grain size, fraction of DRX and MDRX. The equations were implement into a finite element model (FEM) to simulate the evolution of grain structure in the process of two-step blade forging of alloy 718. Gu et al. [14] investigated the effect of temperature, strain rate and pre-strain on the metadynamic softening and recrystallized grain size of Nimonic 80A through interrupted hot compression tests, and predicted the MDRX kinetics using a JMAK model. Zhang et al. [15] used a similar approach to study and model the kinetics of MDRX in Hastelloy C-276, a Ni–Cr–Mo-based superalloy. In consideration of the discontinuous yielding, Lin et al. [2] proposed a

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new approach called maximum stress method to evaluate the MDRX softening fraction of a typical Ni-based superalloy, and the JMAK model was employed to describe the kinetics of MDRX. Recently, Zouari et al. [1] used a two-site mean field model to predict the recrystallized grain size and the recrystallized fraction (RX) of Inconel 718 in both the dynamic and metadynamic regimes and established a connection between the two regimes. In addition to grain refinement, the MDRX can also bring about the evolution of grain boundary, which is benefit for improving the material properties through the grain boundary engineering (GBE). Souaï et al. [16] investigated the grain boundary evolution through hot working and subsequent supersolvus annealing of a Ni-based superalloy PER 72. They found that more $\sum 3$ boundaries were created during supersolvus annealing treatment in the microstructures retaining higher dislocation density after deformation. Through the EBSD analysis, Cao and Di [17] studied the grain boundary character distribution in Incoloy 800 H during SRX and MDRX. It was found that the $\sum 3$ regeneration was the main mechanism for the increase in $\sum 3^n$ boundaries, while new twinning was predominant when MDRX and SRX took effect simultaneously.

The MDRX softening behavior was usually investigated via double hit compression tests and described using empirical equations such as JMAK model and the modified versions in the above studies. However, the recrystallized microstructure, which affects the properties of the products, cannot be fully reflected by the softening fraction during MDRX and the evolution of grain orientation and twin boundaries during MDRX of Ni-based superalloy were rarely studied. Furthermore, the DRX and MDRX are two consecutive processes, and thus, the dynamic recrystallized microstructure could significantly affect the microstructure evolution during MDRX. This fact has often been neglected in previous studies. Therefore, the present work aims to investigate the evolution of microstructure and twin boundaries during MDRX of a Ni-based superalloy using the EBSD technique. Additionally, this work attempts to develop a dislocation density-based model which is capable of predicting the continuous microstructure evolution during DRX and MDRX. First, isothermal compression tests followed by holding at the deformation temperature for different times were carried out. Then, the effect of holding time, temperature and strain rate on the evolution of microstructure and twin boundaries was investigated by EBSD analysis. Finally, a dislocation density - based MDRX model for the studied superalloy was developed, and the reliability of the model was verified based on the experimental recrystallized fraction.

2. Experimental details

The chemical composition (wt%) of the studied superalloy is: 53.73Ni–18.02Cr–5.40Nb–2.88Mo–1.00Ti–0.50Al–0.05Mn–0.025C–(bal.) Fe. Cylindrical test specimens with a diameter of 8 mm and a length of 12 mm were machined from as-received wrought billet. All the specimens were solution treated at 1373 K for 30 min followed by water quenching. In order to study the MDRX behavior, isothermal uniaxial compression tests followed by holding at the deformation temperature for different times were conducted on a Gleeble 1500D thermo-mechanical simulator. Graphite foils were used to reduce the friction between the specimens and the dies. The specimens were heated to the deformation temperature at a rate of 20 K/s and then held for 3 min to ensure temperature uniformity before deformation. Each specimen was deformed to a true strain of 0.357 (higher than the critical strain for DRX) at various temperatures and strain rates. Then, the specimens were kept on the test machine at the same temperature for different holding time before water quenching. The conditions used to analyze the effect of temperature, strain rate and holding time are listed in Table 1.

Table 1
The conditions analyzed for MDRX of the studied superalloy.

Affecting factors	Deformation conditions	Holding time
Temperature	$T=1223$ K, $\dot{\varepsilon}=0.01$ s^{-1}, $\varepsilon=0.357$ $T=1273$ K, $\dot{\varepsilon}=0.01$ s^{-1}, $\varepsilon=0.357$ $T=1323$ K, $\dot{\varepsilon}=0.01$ s^{-1}, $\varepsilon=0.357$ $T=1373$ K, $\dot{\varepsilon}=0.01$ s^{-1}, $\varepsilon=0.357$	0, 5, 30, 60 s
Strain rate	$\dot{\varepsilon}=0.01$ s^{-1}, $\varepsilon=0.357$, $T=1323$ K $\dot{\varepsilon}=0.1$ s^{-1}, $\varepsilon=0.357$, $T=1323$ K $\dot{\varepsilon}=1$ s^{-1}, $\varepsilon=0.357$, $T=1323$ K	0, 5, 30, 60 s

The water quenched specimens were sectioned along the axial direction for microstructure observation. The samples were mechanically polished and then polished electrolytically in a 20% $H_2SO_4+80\%$ CH_3OH solution at 15 V for 40 s at room temperature. EBSD measurement was carried out on a ZEISS ULTRA 55 scanning electron microscope equipped with HKL Channel 5 software. The recrystallized grains can be identified by their small size and equiaxed shape [1]. Average recrystallized grain size was determined using the line intercept method.

3. Results and discussion

3.1. Initial microstructure

The orientation image microscopy (OIM) map and the misorientation angle (θ) distribution of the specimen after solution treatment are shown in Fig. 1, in which the LAGBs ($3° \le \theta < 15°$) and HAGBs ($\theta \ge 15°$) are indicated by gray and black lines, respectively. The majority of the grain boundaries are HAGBs, and the fraction of LAGBs is as low as 2%. The average grain size and mean misorientation angle ($\bar{\theta}$) are determined to be 95 μm and 49.54°, respectively.

3.2. MDRX kinetics and microstructure evolution

3.2.1. Effect of holding time

Fig. 2 shows the OIM maps and misorientation distribution of samples deformed to a strain of 0.357 at 1323 K with strain rate of 0.1 s^{-1} followed by holding at this temperature for different times. A partially recrystallized necklace microstructure is observed in the sample deformed to a strain of 0.357 with no further holding (Fig. 2a). The small recrystallized grains delineated by HAGBs decorate on the old bulging grain boundaries, especially on triple junctions, indicating the initiation of DRX owing to the strain-induced boundary migration [18]. Meanwhile, some LAGBs that produced due to dislocation generation and dislocation boundary formation, are detected mainly at the vicinity of original grain boundaries. After annealing for 5 s (Fig. 2b), the recrystallized grains obviously grow up and expand into the unrecrystallized area, resulting in an apparent increase in fraction of recrystallization and HAGBs. As shown in Fig. 2c, with further annealing, the recrystallized grains become more and more prevalent, and the pre-elongated grains are replaced by the equiaxed grains. The sample almost fully recrystallized when the holding time increases to 60 s, which can be seen in Fig. 2d. Moreover, as the MDRX progresses, the LAGBs gradually disappear, and the HAGBs become dominant. Only a few LAGBs are observed in Fig. 2d, for the sample annealed at 1323 K for 60 s. The average recrystallized grain size at the holding time of 0, 5, 30 and 60 s can