



Effects of initial microstructures on serrated flow features and fracture mechanisms of a nickel-based superalloy

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ABSTRACT

The uniaxial tensile experiments are performed on a nickel-based superalloy with different initial microstructures at intermediate temperatures (473–973) K and low strain rates (0.0001–0.001) s⁻¹. Effects of initial microstructures on plastic deformation mechanisms, serrated flow features, and fracture characteristics are analyzed. It is clearly demonstrated that the plastic deformation mechanism of serrated flow is irrelevant to the initial microstructures, and the dislocation cross slip in the matrix is responsible for the serrated flow. However, the effects of initial microstructures on fracture mechanisms are obvious, i.e., the main fracture mechanism of the ST (solution treated) and HS (solution plus γ'/γ'' phases aging precipitation) superalloys are the nucleation and growth of micro-voids, which accelerates the break of carbides and interfacial failure of carbides/matrix. While for the SAT (solution plus δ phases aging precipitation) superalloy, δ phases and carbides play a significant role in the nucleation of micro-voids. Furthermore, the increase of the deformation temperature reduces the elongation to fracture of the ST and HS superalloys. However, the better tensile ductility is obtained with raising the deformation temperatures for the SAT superalloy.

1. Introduction

Nickel-based superalloys, which have marvelous performances including outstanding corrosion resistance and excellent high-temperature properties, are extensively used in aerospace and nuclear industries [1–5]. However, it is commonly accepted that the serrated flow or Portevin-Le Chatelier (PLC) effect usually occurs in nickel-based superalloys at service temperatures [6–10]. Several investigators demonstrated that the appearance of serrated flow can lead to the inhomogeneous deformation with localization bands [11, 12] and surface roughening [13, 14]. Meanwhile, this phenomenon can result in strain staircase behaviors in the macroscopic time domain [15], and induce the formation and propagation of shear bands in the space domain [16]. Prasad et al. [17] discussed the influences of serrated flow on the low-cycle fatigue properties in a near α Ti alloy. Choudhary et al. [18] found that PLC effect greatly affects the hot ductility and fracture behavior of P92 Steel. Schneider et al. [19] studied the interaction between the serrated flow and surface quality of AA5182 alloy. To represent the serrated flow features of AA5083 alloy, the suitable models were developed by Clausen et al. [20] and Sheikh et al. [21]. Also, Gupta et al. [22] and Garg et al. [23] developed the suitable constitutive models for

describing the DSA behaviors in an austenitic stainless steel.

Generally, in nickel-based superalloys, the complex precipitates including a primary strengthening phase (γ'' , Ni₃Nb), a secondary strengthening phase (γ' , Ni₃Al), an equilibrium phase (δ , Ni₃Nb) and a fine carbide particle play important roles on mechanical properties [24–28]. Also, it is well known that the microstructures are greatly affected by the deformation processes for superalloys [29, 30]. In recent years, some scholars have investigated the influences of different microstructures on the serrated flow of some superalloys. For example, Cai et al. [31] found that γ' precipitates can facilitate the dynamic strain aging effect in the tensile process of Ni-based superalloy. Yuan et al. [32] demonstrated that the mechanical properties and PLC effect are significantly affected by ordered precipitates in Ni-Cr-Mo alloys with different heat treatments. Sundararaman et al. [33] investigated that the effects of aging time on the serrated flow of IN718 alloy, and summarized the regime for the occurrence of serrations at elevated temperatures. Cui et al. [34] found that the mechanical properties and serrated flow features are greatly affected by γ' precipitates in a Ni-Co-Cr-base superalloy. Li et al. [35] studied the influences of annealing processes on PLC effect in a Ni-based superalloy, and revealed that the larger grain size easily induce the serrated flow. Also, Chandravathi

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Table 1
The heat treatment processing.

Case ID	Heat treatments
ST	1313 K × 0.75 h/AC
HS	1313 K × 0.75 h/AC + 993 K × 8 h/FC + 893 K × 8 h/AC
SAT	1313 K × 0.75 h/AC + 1173 K × 8 h/AC

Note: AC represents the air cooling to the room temperature, while FC indicates the furnace cooling to 893 K at 50 K/h.

et al. [36] discussed that the effects of microstructures on hardness, tensile strength and the critical strain for initiating serration flow of a modified 9Cr–1Mo steel. Shen et al. [37] suggested that the serrated flow is greatly affected by different precipitates in 2090 Al–Li alloy. Therefore, the effects of initial microstructures on the serrated flow features in alloys are very complex, which need to be systematically investigated.

Although some researchers have discussed that the effects of microstructures on serrated flow features and mechanical properties of some superalloys, few considerations are paid to the effects of initial microstructures, especially different precipitates, on the plastic deformation mechanisms, serrated flow features, and fracture characteristics at intermediate temperatures and low strain rates. In this study, uniaxial tensile experiments were performed on a nickel-based superalloy with different initial microstructures at 473–973 K and low strain rates (0.0001–0.001) s⁻¹. The influences of initial microstructures on the plastic deformation mechanisms, serrated flow features and fracture characteristics were discussed.

2. Material and Tests

The experimental material employed in present research is a commercial superalloy (GH4169) with alloying elements (wt%) of 52.82Ni - 18.96Cr - 5.23Nb - 3.01Mo - 1.00Ti - 0.59Al - 0.03C - 0.01Co - (bal.)Fe. The dumbbell-shaped tensile specimens were made according to ISO 6982-2 [38], and the dimension of gauge section is 6 mm in diameter and 30 mm in length. To comprehensively investigate the influences of initial microstructures on the plastic deformation mechanisms, serrated flow features, and fracture characteristics, three kinds of heat treatment procedures including ST (solution treated), HS (solution plus γ'/γ'' phases aging precipitation), and SAT (solution plus δ phases aging precipitation) were carried out before uniaxial tensile experiments. Here, the solution plus γ'/γ'' phases aging precipitation is usually named as high-strength processing for the studied superalloy [39]. Therefore, “solution plus γ'/γ'' phases aging precipitation” is written as HS for short. In order to distinguish ST and HS treatments, “solution plus δ phases aging precipitation” is written as SAT for short [40]. Additionally, for the SAT treatment, the aging at 1173 K is widely used for the precipitation of δ phases [3, 4]. Table 1 shows the detailed heat treatment procedures. The typical initial microstructures were analyzed by transmission electron microscopy (TEM) and optical microscopy (OM), as shown in Fig. 1. Obviously, the dislocations appear in planar arrays in the ST superalloy, which contributes to the generation of thermal stresses [33]. In the HS superalloy, there are some homogeneous-distributed ellipsoidal γ' (Ni₃Al) phases and granular γ'' (Ni₃Nb) phases, which can strengthen the alloy. While for the SAT superalloy, lots of orthorhombic δ phases (long needle-like) are observed throughout the matrix. Also, three kinds of microstructures contain some fine inclusions and carbides in the matrix due to smelting process

of this alloy.

Uniaxial tensile tests were conducted on a MTS-GWT2105 machine at (473–973) K and low strain rates (0.0001–0.001) s⁻¹. The detailed uniaxial tensile experimental schedules are presented in Fig. 2. Prior to loading, all the deformed specimens with different initial microstructures were soaked at the tensile temperature for 10 min. During the tensile experiments, the fluctuation of tensile temperature was controlled within 1 K, and the experimental data were automatically recorded by the computer. To investigate the plastic deformation mechanisms of the studied superalloy, the foils for TEM observations were cut from the deformed specimen perpendicular to the tensile axis. Subsequently, 50 μ m-thick foils were prepared by mechanical polishing and electro-polished with a solution consisting of 10% HClO₄ and 90% CH₃CH₂OH (vol%) at 248 K and the current of 50–60 mA. The TEM observations were performed on a Tecnai G2 F20 operating at 200 kV. Meanwhile, all the fracture morphologies were observed using scanning electron microscopy (SEM).

3. Results and Analysis

3.1. High-temperature Tensile Deformation Characteristics

Fig. 3 presents the flow characteristics of the superalloy with different initial microstructures at 0.0003 s⁻¹. It is revealed that the superalloy with different initial microstructures exhibits different hot tensile deformation behaviors. Obviously, a sudden rise in the flow stress before yielding can be observed in the elastic deformation process, which accords with the Hooke's law. Then, due to the accumulation of dislocations, the dislocation multiple sliding appears [25]. Thus, the flow stresses approximately linearly increase to peak values, indicating the nearly linear hardening happens in the plastic deformation process. Finally, the flow stresses decrease until the final rupture of specimens owing to the localized necking. Meanwhile, a large amount of serrations are found from the flow stress curves, which implies the occurrence of serrated flow in the superalloy during the hot tensile. Similar features were also found in other nickel-based superalloy [31, 41]. Also, according to the morphological characteristics, magnitudes and periodicities of serrations, these serrations can be classified into the following four types, i.e., type A serrations are commonly observed in the lower deformation temperature range, and its morphological characteristics are above the general flow curves. Meanwhile, the morphological features of type A + B / B serrations are above or below the general flow curves, which can be easily found at intermediate deformation temperatures. Furthermore, the serrations of type C usually occur at the higher deformation temperatures, and their characteristics are below the general true stress-strain curves. By comparing Fig. 3a–c, it can be noticed that the regimes for serrations not only depend on the deformation conditions, but also relate to the initial microstructures. For example, when deformation conditions are 473 K and 0.0003 s⁻¹, the serrated flow only occurs in the ST or SAT superalloys. However, as the strain rate is 0.0003 s⁻¹, the serrated flow disappears in the SAT superalloy once the deformation temperature exceeds 873 K. Besides, the ultimate strength decreases with increasing deformation temperature due to the thermal softening. This experimental result is in an agreement with other report on nickel-based superalloys [31]. Because of the occurrence of typical work hardening-dynamic recovery phenomenon [1, 27, 42] and the generation of strengthening phases in the matrix at low strain rates [27, 43, 44], the flow stress curves at 973 K are different from those at other temperatures.

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