



# Work-hardening behaviors of typical solution-treated and aged Ni-based superalloys during hot deformation



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## ABSTRACT

A detailed analysis of work-hardening behavior is useful to optimize the hot forming processing of metals or alloys. The hot compression tests of solution-treated and aged Ni-based superalloys are carried out over wide ranges of temperature and strain rate. Based on the experimental data, the effects of strain rate, deformation temperature, strain and  $\delta$  phase ( $\text{Ni}_3\text{Nb}$ ) on the work-hardening behaviors are discussed in detail. The microstructures of the studied superalloy are observed to correlate with the complex dynamic deformation mechanisms. The experimental results show that the work-hardening behaviors of the studied superalloy are significantly affected by deformation temperature, strain rate, strain and  $\delta$  phase. The work-hardening rate increases with the decrease of deformation temperature or the increase of strain rate. When the deformation degree is relatively small,  $\delta$  phase can inhibit the dislocation movement and enhance the work-hardening behavior. However, with further straining,  $\delta$  phase stimulates the occurrence of dynamic recrystallization and promotes the dynamic softening. When the deformation temperature is higher than the dissolution temperature of  $\delta$  phase, the effects of  $\delta$  phase on work-hardening behaviors are weakened.

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## 1. Introduction

Due to their excellent mechanical properties under high temperature, Ni-based superalloys are widely used in aviation and aerospace industries [1]. Generally, Ni-based superalloys are precipitation strengthen alloys, and the strengthening is mainly attributed to  $\gamma'$  ( $\text{Ni}_3(\text{Al,Ti})$ ) and  $\gamma''$  ( $\text{Ni}_3\text{Nb}$ ) phases [2,3]. The  $\delta$  phase ( $\text{Ni}_3\text{Nb}$ ), a equilibrium phase of  $\gamma''$  phase, has high stability below its dissolution temperature [4–6]. It is well known that the final mechanical properties of Ni-based superalloys depend not only on the processing parameters (deformation temperature, strain rate, deformation degree, etc.), but also on the initial microstructures (grain size, second phases, etc.). Therefore, in order to obtain the optimal mechanical properties of workpieces, it is important to study the flow behaviors and microstructural evolution during the hot deformation of Ni-based superalloys [7].

In recent years, many investigators have studied the flow behaviors and microstructural evolution of Ni-based superalloys

[8–35]. Lin et al. [8,9], Ning et al. [10], Yu et al. [11], Yao et al. [12], Wu et al. [13], and Etaati et al. [14] studied the hot deformation behaviors, and developed the suitable constitutive models for some typical superalloys. Based on the results from the isothermal compression or tensile experiments, Wen et al. [15], Wang et al. [16], Ning et al. [17], and Cai et al. [18] constructed the processing map for different superalloys, and the optimal working domains were indentified. Chen et al. [19], Shore et al. [20], Tian et al. [21], Qi et al. [22], Dehghani et al. [23], Cheng et al. [24], Guo et al. [25], Lee and Hou [26], and Wang et al. [27] discussed the microstructural evolution mechanisms, such as discontinuous and continuous dynamic recrystallization, of some typical superalloys during the hot deformation. The precipitation mechanism of  $\eta$  phase ( $\text{Ni}_6\text{AlNb}$ ) [28], phase transformation [29], the coarsening of strengthening phases ( $\gamma'$  and  $\gamma''$ ) [30], and strengthening mechanism [31] of Inconel 718 superalloy were studied. Additionally, the effects of  $\delta$  phase on the hot deformation behaviors of Inconel 718 superalloy were analyzed by Lin et al. [32,33], Wang et al. [34], and Thomas et al. [35].

It is well known that a detailed analysis of the work-hardening behavior is useful to optimize the hot forming processing of metals or alloys. Previously, several researches were conducted on the

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work-hardening behaviors of different materials [36–48]. Puchi-Cabrera et al. [36] employed the differential form of the phenomenological exponential-saturation work-hardening law to express the work-hardening rate as a function of the yield and saturation stresses, as well as the athermal work-hardening rate and flow stress itself, for a C–Mn steel. Also, Puchi-Cabrera et al. [37] employed a simplified approach based on the Mechanical Threshold Stress (MTS) model to describe the flow behaviors of 20MnCr5 steel, considering the evolution of the flow stress due to work-hardening. Also, the work-hardening behaviors of Ni–42.5Ti–7.5Cu alloy [38], a high Nb containing TiAl alloy [39], pure Ti [40], a low carbon vanadium-nitride microalloyed steel [41], hot-rolled AZ31 magnesium alloy [42], Al–7%Si–1%Cu–0.5%Mg alloy [43], Ni-based superalloy [44], AA1070 aluminum alloy [45], Al–5356 alloy [46], AA6111 and AA7030 aluminum alloys [47], and 304 stainless steel [48] were investigated.

From the previous literatures [36–48], it can be found that the work-hardening behaviors and the deformation mechanisms are very complicated. However, the investigations focusing on the work-hardening behavior of Ni-based superalloy during hot compressive deformation are seldom, especially the effects of complex precipitates on work-hardening behaviors. Therefore, further analysis should be carried out to comprehensively understand the work-hardening behaviors of Ni-based superalloys under hot working conditions. In this study, the work-hardening behaviors of a Ni-based superalloy are investigated by hot compression tests over wide ranges of deformation temperature and strain rate. Based on the experimental results, the effects of deformation temperature, strain rate, strain and  $\delta$  phase on the work-hardening behaviors are discussed in detail. The deformed microstructures are observed to correlate with the experimental phenomenon and theoretical analysis.

## 2. Materials and experiments

The material used in this investigation was a typical Ni-based superalloy, and its chemical compositions (wt%) are 52.82Ni–18.96Cr–5.23Nb–3.01Mo–1.00Ti–0.59Al–0.01Co–0.03C–(bal.)Fe. Cylindrical specimens with diameter of 8 mm and height of 12 mm were machined from billets. The specimens were solution treated under 1040 °C for 45 min, and then immediately quenched by water. In order to investigate the effects of  $\delta$  phase on the work-hardening behaviors of the studied superalloy, some solution treated specimens were aged under 900 °C for 9 h, and then quenched by water. Hot compression experiments were performed on Gleeble 3500 thermo-mechanical simulator under the deformation temperatures of 920, 980 and 1040 °C, and strain rates of 0.001 and 1 s<sup>-1</sup>. Prior to loading, the specimens were heated to the deformation temperature at a heating rate of 10 °C/s, and then held for 5 min to eliminate the thermal gradient. After the hot deformation, the specimens were immediately quenched by water.

The microstructures of deformed specimens were observed by optical microscope (OM) and transmission electron microscopy (TEM). For OM analysis, the deformed specimens were sliced paralleling to the axial section, and then the exposed surfaces were polished and etched in a solution consisting of HCl (100 ml), CH<sub>3</sub>CH<sub>2</sub>OH (100 ml), and CuCl<sub>2</sub> (5 g) at room temperature for 3–5 min. The microstructures of deformed specimens are observed by Leica optical microscope. For TEM analysis, the thickness of foils sliced from the deformed specimens were grinded to 70  $\mu$ m, and then electro-polished using a solution of 10% HClO<sub>4</sub> and 90% CH<sub>3</sub>CH<sub>2</sub>OH. The foils were examined by JEM-2100F microscope operating at 200 kV. Fig. 1 shows the microstructures of the solution-treated and aged superalloys. It can be found that there are no marked changes in grain size, and the mean grain size is about 75  $\mu$ m. Fig. 1(a) shows that the microstructure is composed of fine equiaxed grains and a great number of straight annealing twins. While for the aged superalloy, a great number of needle-shaped  $\delta$  phases distribute in the grain interior and boundaries (Fig. 1b).

## 3. Results and discussion

### 3.1. Dynamic flow behaviors of the studied superalloy

The following sections will discuss the hot deformation characteristics of the solution-treated and aged superalloys, and then

analyze the effects of  $\delta$  phase on the high-temperature deformation behaviors.

Fig. 2 shows the true stress–true strain curves of the solution-treated and aged superalloys before the peak strain, i.e., the strain corresponding to the maximum stress. It can be found that the flow stress is significantly affected by the deformation temperature, strain rate and strain. With the increase of deformation temperature or the decrease of strain rate, the flow stress decreases obviously. The flow stress increases rapidly till a peak value under a small strain, showing a typical work hardening–dynamic recovery (WH–DRV) stage [49,50]. From Fig. 2, it can also be found that the WH–DRV stages under low deformation temperature or high strain rate are longer than those under high deformation temperature or low strain rate. In other words, the dynamic recrystallization proceeds slowly under low deformation temperature or high strain rate. On the one hand, the low temperature cannot offer sufficient energy for the mobility of grain boundaries, as well as for the nucleation and growth of dynamically recrystallized grain. Meanwhile, the deformation is too quick for the growth of dynamically recrystallized grains when the strain rate is too high. Additionally, Young's moduli can indicate the ability to resist elastic deformation. The underlying reason for Young's modulus difference between the solution-treated and aged superalloys can be attributed to the existence of  $\delta$  phase. Generally,  $\delta$  phase in the aged superalloy can impede the dislocation slip to some extent. The moving dislocations pile up in the vicinity of  $\delta$  phase, and increase the deformation resistance. Therefore, Young's moduli and flow stress of the aged superalloy are higher than those of the solution-treated superalloy, as shown in Fig. 2a.

From Fig. 1, it has been found that the heat treatments significantly affect the precipitation of  $\delta$  phase. So, the effects of heat treatments on the work-hardening behaviors are obvious, as shown in Fig. 2. Compared with the solution-treated superalloy, the peak strain of the aged superalloy is relatively small, except under the deformation temperature of 1040 °C. The main reason for this phenomenon is that  $\delta$  phase can accelerate the dislocation generation and stimulate the occurrence of dynamic recrystallization, if the deformation temperature is below the dissolution temperature of  $\delta$  phase (about 1038 °C). When the deformation temperature is 1040 °C,  $\delta$  phase quickly dissolves during hot deformation. The dissolution of  $\delta$  phase improves the compatibility of matrix and  $\delta$  phase, which slows down the rate of dislocation generation. Therefore, the work-hardening behavior caused by  $\delta$  phase is weakened under the deformation temperature of 1040 °C.

Fig. 3 shows the peak stresses of the solution-treated and aged superalloys under the tested conditions. It can be found that the peak stresses of the aged superalloy are higher than those of the solution-treated superalloy, except those under the deformation temperatures of 920 °C and 980 °C and strain rate of 1 s<sup>-1</sup>. According to the previous analysis [33],  $\delta$  phase can significantly strengthen the matrix. However, when the deformation temperatures are 920 °C and 980 °C, the peak stresses of the aged superalloy are low under the high strain rates. Due to the incompatibility of  $\delta$  phase and matrix,  $\delta$  phase can impede the dislocation slip. The moving dislocations are piled up in the vicinity of  $\delta$  phase, leading to the stress concentration. Under the high strain rate, the enhanced stress concentration easily causes the interfacial debonding between  $\delta$  phase and matrix, as well as the brittle fracture of  $\delta$  phase. Therefore, the flow stress decreases, and the high strain rate is not suitable for the precision forming of this superalloy [15].

### 3.2. Work-hardening behaviors of the solution-treated superalloy

During hot forming, the work-hardening behaviors of metals or alloys are often very complicated [42,51]. Generally, the work-hardening behaviors are investigated by analyzing the variations

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