Mechanical Behavior and Microstructure Evolution during Cold Deformation and Solution Treatment of IN718 Alloy

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Abstract. IN718 alloy possesses excellent mechanical properties from room temperature to 650 °C and good processability; therefore, it has been widely used in turbine-engines and aircraft fasteners. Work hardening is an important strengthening method for this alloy. In this paper, the cold deformation behavior of IN718 alloy, including stress-strain curves, the constitutive relationship, and the microstructural evolution were investigated through room-temperature compression testing with strain rates from 0.1~1 s\textsuperscript{-1} and engineering strains from 10% ~ 40%.

Introduction

IN718 alloy is a precipitation-hardened nickel-iron base alloy, containing significant amounts of chromium, niobium, and molybdenum, and lesser amounts of aluminum and titanium. It finds extensive application in the aerospace and petrochemical industries because of an excellent combination of strength, corrosion resistance, and processability. IN718 alloy offers exceptionally high strength potential for application to 650 °C and excellent weldability due to the slow precipitation of the strengthening phase. Because of these attractive properties, this alloy has been extensively used in gas turbine engines as discs, blades, shafts, casings, fasteners, thrust reversers, etc. [1-5].

Superalloy fasteners are important bearing connectors for aerospace engines and aircraft. They are mainly used for the connection of high-speed rotary compressors and turbine rotors, aircraft wing fuselage and other key parts. They must have high tensile strength; excellent overall properties such as shear strength, high durability and creep resistance, fatigue resistance, stress relaxation resistance, excellent corrosion resistance; and a matched thermal expansion coefficient. Rahimi et al. (2017) demonstrated that the IN718 alloy has excellent stress relaxation properties after ageing heat treatments[3]. Du et al. (2007) studied the high temperature structure stability and mechanical properties during long-term thermal exposure\textsuperscript{[4]}\textsuperscript{[4]}. Lu et al. (2014) found that the Larson-Miller relationship was obeyed during high temperature stress rupture[5]. Lu et al. (2002) studied the relationship between the fine-grained pretreatment process and superplastic deformation[6]. IN718 alloy possesses excellent mechanical properties from room temperature to 650 °C and good processability, therefore, it has been widely used in aero engine and aircraft fasteners.

Hot working is a shaping process that is widely employed in metallic materials. Thomas et.al (2006) confirmed that forming at high temperature involves large strain at relatively low stresses, but (at the same time) heavy modifications of the microstructure occur[7]. Rao et al. (2004) investigated the microstructure and mechanical properties of as-HIPed and HIP+ heat-treated IN718 alloy after thermomechanical working[8]. To control every step of the thermomechanical processing and thereby obtain the most favorable mechanical properties and microstructures, attention has been paid in past decades to the hot deformation behavior and computer simulation of thermomechanical processing of IN718 alloy. Praveen et al. (2008) found that the work-hardening did something or other regularity during hot deformation\textsuperscript{[1]}\textsuperscript{[1]}. Coste et al. (2005) discovered that the varying phase fraction can significantly affect the microstructure and mechanical properties during
and after the deformation process[2]. Kashyap and Chaturvedi (2007) examined that the effect of prior annealing on high temperature flow properties and microstructural evolution[9]. Azarbarnas et al. (2016) analyzed the dynamic recrystallization mechanisms and twining evolution during hot deformation[10]. Kumar et al. (2017) examined the work hardening characteristics and microstructural evolution at moderate strain rates[11]. Zhang et al. (1997, 1999 and 2000) analyzed the strain-rate hardening behavior and constructed a model of the grain structure during hot deformation[12-14]. Na et al. (2003) simulated the microstructure of an Alloy 718 blade forging using 3D FEM[15]. Yeom et al. (2007) predicted the microstructural evolution in the cogging of an Alloy 718 ingot[16]. IN718 is very sensitive to hot deformation conditions and thermomechanical processing. In particular, the deformation temperature and strain rate will severely affect the flow stress and microstructure of IN718 alloy. However, there are still only a few reports in the literature so far concerning the cold deformation behavior of IN718. In contrast to high temperature deformation, cold deformation at room temperature involves little strain at relatively high stresses. The aim of the present work was to study the cold deformation flow behavior of a commercial IN718 alloy, analyzing the work-hardening law and microstructure evolution mechanism under these conditions.

Experimental Procedures

The IN718 alloy used in this work was obtained by vacuum induction melting (VIM) and vacuum arc remelting (VAR). After homogenization, the remelted ingot was hot rolled into a 26 mm diameter bar. The chemical composition of the IN718 alloy selected is listed in Table 1. Noticeable amounts of Nb, Ti, and Al are present, as well as some Co.

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Room temperature uniaxial compression tests were carried out on the MTS 810 to evaluate the cold deformation behavior. Cylindrical samples, 8 mm in diameter and 12 mm in length, were machined for this purpose. They were tested at three different strain rates: 0.1, 0.5, and 1 s\(^{-1}\), at four deformation amounts: 10\%, 20\%, 30\%, and 40\%. Metallographic samples were prepared using standard mechanical polishing procedures and were electrolysically etched in a mixture of 150 mL H\(_3\)PO\(_4\), 10 mL H\(_2\)SO\(_4\), and 15 g chromic anhydride (CrO\(_3\)). The microstructural characteristics of the alloy were investigated using optical microscopy (OM) and scanning electron microscopy (SEM). A JSM-7800F type electron backscatter diffraction (EBSD) detector was used to analyze the deformation mechanisms of the room temperature compression specimens.

Results and Discussion

True Stress-True Stain Curves

Figure 1 shows the true stress-true strain curves for room temperature compression tests of IN718 alloy at various strain rates. As shown in Figure 1, the stress rises sharply with the increase in strain to near the yield stress and then slowly rises to stability. The trend of the stress-strain curve conforms to the rheological characteristics of a low-stacking-fault-energy metal, indicating that the alloy undergoes work hardening during cold deformation. The true stress-true strain curve can be divided into two stages. When the strain is small, the work hardening phenomenon is significant, the work hardening exponent is large, and the curve is roughly straight; this is the linear hardening stage. At the time of work hardening, accompanied by deformation softening, the work hardening exponent decreases as the amount of deformation increases; this is the parabolic hardening stage. when the strain rate is constant, the deformation resistance of the alloy increases as the strain increases. For deformation amounts in the range from 0 to 40\%, the three true stress-true strain curves with different strain rates are basically coincident, indicating that the alloy is only sensitive
to the deformation amount under room temperature plastic deformation conditions, and is not sensitive to the change of the strain rate.

Figure 1. True stress-true strain curves for room temperature compression tests at various strain rates: (a)0.1, (b)0.5, (c)1.0 s\(^{-1}\).

Cold Deformation Constitutive Relationship

The alloy matrix is a face-centered cubic austenite structure with a low stacking-fault energy. The alloy contains a large number of annealed twins, and its constitutive relationship cannot be described by simply using \( \sigma = k \cdot \varepsilon^n \); the more complicated relationship \( \sigma = k \cdot \varepsilon^{n_1+n_2\ln \varepsilon} \cdot \dot{\varepsilon}^m \) is necessary. In this formula, \( k \) is the material-dependent constant, \( n_1 \) and \( n_2 \) are the work hardening exponents, and \( m \) is a strain-rate-sensitive factor. The results of curve-fitting show that the cold deformation constitutive relationship of IN718 alloy is given by the following formula (1):

\[
\sigma = 1178 \cdot \varepsilon^{(-0.016-0.083 \cdot \ln \varepsilon)} \cdot \dot{\varepsilon}^{0.019}
\]  

(1)

In order to further study the effects of cold deformation and strain rate on work hardening, we examined their effects separately. According to the fitted constitutive relationship of cold deformation in IN718 alloy, the work-hardening rate (\( \theta=d\sigma/d\varepsilon \)) can be calculated between 0.1~1 s\(^{-1}\). As shown in Figure 2, when the strain rate is held constant, the work hardening rate of IN718 alloy is significantly reduced with increases in the amount of cold deformation, and then tends to stabilize. When the amount of cold deformation is held constant, the work-hardening rate is basically unchanged with increases in the strain rate. Therefore, the work-hardening rate of IN718 alloy is mainly affected by the amount of cold deformation; and the effects of the strain rate are not significant.
The climb and cross-slip of the dislocations in a low-stacking-fault-energy alloy are impeded when the dislocations encounter a barrier (vacancy, dislocation, grain boundary, etc.). Recovery proceeds slowly; the high density dislocations are intersected and tangled; and then the network structure forms, which makes the slip of the dislocations unlikely. The flow stress significantly increases with the increase of strain, due to dislocation strengthening.

A representative work hardening plot obtained during cold deformation of the studied IN718 alloy is presented in Figure 3. Generally, the $\theta$-\(\varepsilon\) curve of the currently studied alloy reveals two stages: The first stage (stage-I) is characterized by a drastic rise in \(\theta\) with the increase of true stress in a linear fashion, which is an obvious manifestation of the progress of hardening following elastic deformation. The second stage (stage-II) is characterized by a drastic drop in \(\theta\) with increasing true stress in a parabolic fashion. Stage-II extends up to the initiation of sub-grains by the change in the slope of the $\theta$-\(\varepsilon\) curve. Stage-II corresponds to the region dominated by the slip, climb, and cross-slip of the dislocations, prior to the onset of substructure formation. The work hardening rate decreases with the increased strain, because of the annihilation and recombination of dislocations.

**Microstructure Evolution**

Figure 4 shows the grain microstructure under different amounts of cold deformation. It indicates that the amount of cold deformation has a very significant influence on the grain structure. When the amount of cold deformation increases, the degree of deformation of the grains increases, and the deformation uniformity of the grains gradually improves; consequently, the average grain size is reduced.
when the amount of deformation is 10%, the grains are slightly elongated, and a small number of deformed twins appear. When the amount of deformation continues to increase, the grains undergo significant compression deformation, the grains are deformed into a flat shape, and a large number of deformation twins and slip lines appear. When the amount of cold deformation is further increased to 40%, the degree of grain deformation is larger, showing a thin strip; the deformation zone is increased, the slip line is increased, the twin boundary is clearly visible, and the parallel sliding line is terminated at the twin boundary. The twin boundaries and the grain boundaries present a strong hindrance to dislocations. As the amount of deformation increases, the degree of deformation unevenness decreases significantly, the deformation of grains with different orientations becomes more and more uniform, and the average grain size decreases.

The influence of deformation amount on the evolution of grain boundaries is illustrated by orientation imaging microscopy (OIM) maps highlighted with grain boundaries as shown in Figure 5. For the cold deformation samples, the high-angle grain boundaries (HAGBs, θ > 15°), low-angle grain boundaries (LAGBs, 2θ < 15°) and twin grain boundaries are highlighted by solid black, green, and red colored lines respectively. The OIM maps of cold deformation samples at room temperature are dominated by LAGBs when deformed at both strain rates. The distribution of LAGBs, indicates the considerable existence of dislocation density with the grain interior and near grain boundaries.

During the cold deformation, the grains are elongated, forming of dislocation cells and deformation twins, is as obstacles existing to the moving of the dislocations in work hardening. The internal microstructural change of the alloy will directly manifest itself in the external mechanical properties. The specific outcome is that the strength and hardness increase, while the ductility and toughness decrease.

As shown in Figure 6, the fraction of LAGBs significantly increases from 33% in the non-deformed sample to 82% in the 30% cold deformation sample; the fraction of random HAGBs with 10-50° slowly decreases; and the fraction of twin boundaries with 60° misorientation (i.e. from 16% to 3%) slowly decreases. When the amount of deformation exceeds 30%, the fraction of LAGBs shows a downward tendency, and the fraction of HAGBs shows the opposite tendency.

Dislocations of nickel-based IN718 alloy with low-stacking-fault-energy material are incomplete dislocations. The mobility of incomplete dislocations is high at the initial stage of deformation when materials can be deformed through slipping of incomplete dislocations. When the amount of deformation reaches a certain level, with increasing strain rate, the stress becomes easier to concentrate; and more twins will be broken in the deformed grains, leading to a decrease in the number of twins. When the deformation amount exceeds 30%, the fraction of the LAGBs decreases accompanied by a decrease of dislocation density and the formation of sub-grains.
Figure 5. EBSD maps showing the grain boundary distribution at a strain rate of 0.1 s⁻¹, for various amounts of deformation: (a)0%, (b)10%, (c)20%, (d)30% and (e)40%.

Green: low-angle GB (2~15°)
Black: high-angle GB (15~55°)

Figure 6. Grain boundary distributions at a strain rate of 0.1 s⁻¹ for various amounts of deformation: (a)0%, (b)10%, (c)20%, (d)30% and (e)40%.

Effect of Solution Treatment

After cold working, superalloys need solution treatment to eliminate residual stress and achieve recrystallization. For the cold-deformed IN718 alloy, the deformation storage energy retained in the alloy during cold deformation includes not only the distortion energy caused by the high-density dislocation accumulation near the grain boundaries, but also the distortion energy caused by the many deformation twins and the energy increase that results from the interactions between the dislocations and the twins. These unstable structures and energy states are eliminated by annealing, which restores the processing plasticity of the IN718 alloy.

As shown in Figure 7, after solution treatment at 960 °C for 1 h, the microstructure of the cold deformation samples changes significantly (except for the 10% deformation sample). In the other three cases, the fraction of LAGBs decreases significantly, and the fraction of HAGBs increases significantly. Under solution treatment, dislocations were active in the sample with 10%
deformation; the dislocations proliferate by slip and climb motion. In nickel-based alloys with low stacking-fault energy, the incomplete dislocation motion produces a stacking fault and a large number of annealed twins; after solution treatment at 960 °C for 1 h, the recovery process is completed, and the dislocation density is reduced significantly, as shown in Figure 7 (b).

Figure 7. EBSD maps showing the grain boundary distribution at a solution temperature of 960 °C for 1 h with various amounts of deformation: (a)10%, (b)20%, (c)30%, and (d)40%.

As shown in Figure 8, after solution treatment at 960 °C for 1 h, the fraction of LAGBs decreases significantly from 72% in the 10% deformation sample to 15% in the 40% deformation sample; the fraction of random HAGBs with 15 ~ 55° increases by rapidly from 20% in the 10% deformation sample to 65% in the 40% deformation sample; and the fraction of twin boundaries increases at first and then decreases slowly. The fraction of twin boundaries in the 20% deformation sample is the highest, reaching 37%. When the amount of deformation exceeds 30%, the total combined fraction of HAGBs and twin boundaries is above 80%, indicating that static recrystallization is complete.

Figure 8. Grain boundary distribution at a solution temperature of 960 °C for 1 h (strain rate 0.1 s-1)

With an increase of solution treatment temperature, the motion of the dislocations is accelerated, leading to the annihilation and recombination of dislocations and the elimination of deformation bands and residual stresses inside the grains after cold deformation. By enough energy entry overcome the energy barrier between metastable state and stability, materials are soft through recovery and static recrystallization. As shown in Figure 9, when the solution treatment temperature is between 950 and 970 °C, the total combined fraction of the HAGBs and the twin boundaries is close to 80% in the case of the 40% cold deformation sample; the dislocation density decreases significantly; and recovery and static recrystallization of the material is complete.
Figure 9. Grain boundary distributions at various solution temperatures for 1 h (deformation amount 40%).

Conclusions
The IN718 alloy is only sensitive to the amount of deformation under room temperature compression conditions, and is not sensitive to changes in the strain rate.

The cold deformation constitutive relationship of IN718 alloy at deformations in the range of 10% ~ 40% and strain rates of 0.1 ~ 1s\(^{-1}\) is:  
\[ \sigma = 1178 \cdot \varepsilon^{(-0.016-0.083 \ln \varepsilon)} \cdot \dot{\varepsilon}^{0.019} \]

The \(\theta-\varepsilon\) curve of the currently studied alloy reveals two stages. The first stage is characterized by a drastic increase in \(\theta\) with the increase of true stress in a linear fashion. The second stage is characterized by a drastic drop in \(\theta\) with the increase of true stress in a parabolic fashion.

Under the same solution treatment conditions, recovery and static recrystallization are more complete with increased amounts of deformation.

References


