High-temperature and room-temperature compression deformation behavior comparing of GH4169 Alloy

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Abstract. The hot deformation characteristics of a Ni-Fe based superalloy (GH4169) were studied in the temperature range 900-1020 °C and strain rate range 0.001-0.1 s⁻¹ using hot compression tests. The cold deformation characteristics of the alloy were studied at room-temperature and strain rates of 0.1-1.0 s⁻¹ using room-temperature compression tests. A hot deformation equation is given to characterize the dependence of peak stress with temperature and strain rate. A cold deformation equation is given to characterize the dependence of peak stress. Comparing the results of the high-temperature and room-temperature deformations of the GH4169 alloy indicated that hot deformation is sensitive to both temperature and strain rate, while the cold deformation is sensitive to strain. At room-temperature deformation conditions, for true strain ≈ 0.2, the dynamic recrystallization nucleates and grains refine.

Keywords: Ni-Fe based superalloy; Deformation; Recrystallization

1. Introduction

The GH4169 alloy is a precipitation-hardened nickel-iron base superalloy, containing significant amounts of chromium, niobium, and molybdenum, and lesser amounts of aluminum and titanium. The alloy finds extensive application in the aerospace and petrochemical industries because of its excellent combination of strength, corrosion resistance, and processability. The GH4169 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].

Hot working is a shaping process that is widely employed in metallic materials. Rao et al. (2004) investigated the microstructure and mechanical properties of as-HIPed and HIP + heat-treated GH4169 alloy after thermomechanical working [6]. To control every step of the thermomechanical process 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 the GH4169 alloy. Praveen et al. (2008) found that the work-hardening behavior not to obey the simple power law relationship either in the solution treated as well as in the different age-hardened conditions [1]. Coste et al. (2005) discovered that varying of the δ 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 [7]. Azarbarmas et al. (2016) analyzed the dynamic recrystallization mechanisms and twinning evolution during hot deformation [8]. Kumar et al. (2017) examined the work hardening characteristics and microstructural evolution at moderate strain rates [9]. Zhang et al. (1997, 1999 and 2000) analyzed the strain-rate hardening behavior and constructed a model of the grain structure during hot deformation [10-12]. Above results confirmed that the GH4169 alloy 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 the GH4169 alloy. However, there are only few reports in the literature concerning the cold deformation behavior of the GH4169 alloy. In contrast, cold deformation at room temperature involves small strains at relatively high stresses. The aim of the present work is to study the cold deformation behavior of the commercial GH4169 alloy, analyzing the work-hardening law and microstructure evolution mechanism under room-temperature deformation conditions.

2. Experimental

The chemical composition of the Ni-Fe based superalloy used in this study is shown in Table 1. Hot compression tests were conducted using a Gleeble 3500 simulator at the temperature of 900 °C, 940 °C, 980 °C, 1020 °C and in the strain rate range of 0.001-0.1 s^{-1} with 12 mm diameter, 18 mm high cylindrical specimens. All specimens were quickly heated to hot deformation temperature and held for about 5 min, and then deformed at true strain of 0.5; immediately, cooling in water until room temperature. Room temperature compression tests were conducted in a Gleeble 1500 simulator at strain rate of 0.1-1.0 s^{-1} and engineering strain of 10 %, 20 %, 30 % and 40 % in cylindrical specimens of 10 mm-diameter and 15 mm-height. The specimens were sectioned parallel to the compression axis and their surfaces prepared for microstructure characterization by EBSD (electron backscatter diffraction). The load-stroke data obtained in compression were processed to obtain true stress-true plastic strain curves using the standard method.

| Table 1. Chemical composition of the GH4169 alloy (wt.%) |
|----------------|---------|----------------|--------|--------|--------|--------|--------|--------|
|                | Al      | C      | Co    | Cr     | Fe     | Mo     | Ti     | Nb     | Ni     |
|----------------|---------|--------|-------|--------|--------|--------|--------|--------|--------|
| Al             | 0.54    | 0.03   | 0.02  | 19.31  | 18.15  | 3.15   | 1.02   | 5.19   | 53.75  |

3. Results and discussion

3.1. True stress-true strain curves and constitutive relationship of hot compression

A series of typical true stress-true strain curves of the GH4169 alloy under thermal deformed conditions (at temperature from 900 °C to 1020 °C and strain rates from 0.001 to 0.1 s^{-1}), is shown in Figure 1. It can be seen that at the initial deformation stage, the rheological stress increases with the increase of strain, and after reaching the peak stress, the rheological stress decreases with the increase of strain. The initial rapid rise in stress is associated with an increase in dislocation density and the formation of poorly developed sub-grain boundaries. For low stacking-fault-energy nickel-based alloys, the dynamic recovery process is inhibited, and when the critical strain is exceeded, the material is softened mainly through dynamic recrystallization.

Peak stress at different temperatures and strain rates can be obtained from the stress-strain curve of alloy GH4169 under isothermal deformation. As can be seen from Figure 2 and Figure 3, there is a classical relationship between peak stress, deformation temperature and strain rate. The peak stress increases with the decrease of deformation temperature, and increases with increasing strain rate. The peak strain also increases with decreasing deformation temperature. The results of curve-fitting show that the hot deformation constitutive relationship of GH4169 alloy is given by the following relation:

\[
\dot{\varepsilon} = 1.58 \times 10^{18} \sigma^{0.17} \exp(-436000/RT)
\]
Figure 1. True stress-true strain flow curves for GH4169 alloy under different strain rates and temperature: (a) 900 °C, (b) 940 °C, (c) 980 °C and (d) 1020 °C.

Figure 2. Relationship between the peak stress and peak strain of GH4169 alloy.

Figure 3. Relationship between the peak stress and temperature of the GH4169 alloy.

3.2. True stress-true strain curves and constitutive relationship of cold compression

Figure 4 shows the true stress-true strain curves for room temperature compression tests of GH4169 alloy at various strain rates. As shown in Figure 4, 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. During work hardening and deformation softening, the work hardening exponent decreases as the amount of deformation increases. This is the parabolic hardening stage.

For constant strain rate, the deformation resistance of the alloy increases as the strain increases. For engineering strain 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 strain under room temperature plastic deformation conditions, and is not sensitive to the change of the strain rate.

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 $\sigma = k \cdot \varepsilon^n$; and a more complicated relationship $\sigma = k \cdot \varepsilon^{n_1+n_2 \cdot \ln \varepsilon} \cdot \varepsilon^m$ is required. Here, $k$ 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 GH4169 alloy is given by the following relation:

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

![Figure 4. 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}.](image)

3.3. Comparing of hot and cold deformation recrystallization behavior

According to the traditional metallography theory, the metal hot deformation is carried out at high temperature, above the recrystallization temperature. In the deformation process, recovery and recrystallization occurs simultaneously. Hot working refers to the deformation process in which the metal hardening rate is equal to its softening rate. The simultaneous recovery and recrystallization during hot deformation are called dynamic recovery and dynamic recrystallization. Dynamic recrystallization usually occurs in materials with low fault energy, such as Cu, Ni and austenite. In the process of hot deformation, the dislocation cross slip and climb of such materials are difficult, and it is not easy to offset the dislocation proliferation by dynamic recovery alone. The storage energy
accumulates to enough high level in each local area to induce recrystallization nucleation. Through the growth of new grains, dislocations disintegrate or disappear in large quantities and clusters, softening the metal and decreasing the deformation stress. As the deformation occurs, work hardening appears and is eliminated by recrystallization, resulting in a wave shape of the stress-strain curve. With the increase of strain, recrystallization occurs successively in different regions of the material, so that the periodicity of overall softening of the material disappears and the stress-strain curve becomes more stable [13-16]. According to literature reports [17], the critical strain of dynamic recrystallization for thermal deformation of the GH4169 alloy increases with the decrease of temperature, from 0.053 at 1066 °C to 0.19 at 927 °C.

![Figure 5. OIM maps showing the grain boundary distribution at a strain rate of 0.1 s⁻¹, for various engineering strain: (a) 0 %, (b) 10 %, (c) 20 %, (d) 30 % and (e) 40 %](image)

Under cold deformation conditions, the influence of strain on the evolution of grain boundaries is illustrated by orientation imaging microscopy (OIM) maps highlighted with grain boundaries as shown in Figure 5. During cold deformation, the grains elongate, forming dislocation cells and deformation twins, and act as obstacles for moving dislocations in the 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 low angle grain boundary (LAGBs) significantly increases from 33 % in the non-deformed sample to 82 % in the 30 % cold deformation sample; the fraction of random high angle grain boundary (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 opposite tendency.
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 %.

When specimens strain from 30 % to 40 %, the fractions of the LAGBs decrease and HAGBs increase. This result shows that dynamic recrystallization occurs at room-temperature. This is completely different from the traditional metallography theory where cold deformation does not occur during dynamic recrystallization.

Dislocation proliferation leads to hardening and dislocation transfer between the slip surfaces through cross slip and climb, resulting in dislocation cancellation and softening. In the initial deformation, the dislocation proliferates rapidly and the deformation stress rises. Until the dislocation density reaches a certain value, the rate at which new dislocations are generated in the material is equal to the rate at which the same number of dislocations disappear. If the dislocation density in the material remains constant, the continuous deformation can be carried out under constant stress. Since the dislocation density of the materials with dynamic recovery no longer increases when the stable stress continues, the deformation storage energy generally cannot accumulate to the extent of inducing recrystallization, so dynamic recrystallization generally does not occur. The microstructure of dynamic recovery is that there are equiaxial subgrains within the fibrous grains, and the dislocation density within the subgrains boundary (or cell wall) remains constant. Such subgrains are formed by repeated multilateralization (or normalization) during dynamic recovery.

Metals with large cold deformation or low stack fault energy tend to form recrystallization cores by subgrain growth. When the deformation strain of metal is large, dislocation density is high and recrystallization driving force is large. When the fault energy is low and the dislocation is in the recovery stage, it is not easy to form a large angle subgrains boundary with high mobility depending on the polylization or subgrains merging mechanism of climbing and cross slip. In the metal under such conditions, firstly, in a small region with a high dislocation density, a subgrains with a low dislocation density can be formed through dislocation movement and rearrangement. The dislocation in this subgrains region is concentrated at the subgrains boundary, which makes the dislocation difference of the subgrain boundary increase, and it is easy to move to the surrounding region with high dislocation density. The dislocation density in the subgrain boundary becomes higher and higher, and the subgrain boundary and the surrounding deformable matrix gradually increase, forming a larger angle subgrain boundary with high mobility, and even some of the subgrain boundaries lose their stability and reconstitute into a larger angle grain boundary, which becomes the recrystallization core.
By comparing to the traditional cold deformation theory, during the cold deformation of the GH4169 alloy, and engineering strain exceeds at 30 %, dynamic recrystallization has already taken place. Further increase of deformation, recrystallization carries out completely. When the engineering strain reaches at 40 %, many non-distorted recrystallization cores form at the original grain boundary, and the grain structure is significantly refined, as shown in Figures 5 and 7.

4. Conclusions

Hot compression tests done at 900-1020 °C and cold compressing tests at strain rates of 0.1-1 s⁻¹ for the GH4169 alloy has been conducted. The following conclusions have been drawn from of results of this investigation.

1) The hot deformation constitutive relationship of the GH4169 alloy is given by:

\[ \dot{\varepsilon} = 1.58 \times 10^{18} \sigma^{6.17} \exp(-436000/RT) \]

2) The cold deformation constitutive relationship of the GH4169 alloy is given by:

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

3) Under cold deformation at strain rates of 0.1-1 s⁻¹ and room temperature, occurrence of dynamic recrystallization tends to be refined grains.

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