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Static Recrystallization Behavior of Inconel 718 Alloy during Thermal Deformation

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Abstract: The softening behavior of Inconel 718 alloy at different temperatures was studied using two-stage interrupted compression method on Gleeble1500D thermal stimulator, and the 2% offset method was applied to analyze the experimental dates. Finally, the static recrystallization fraction was obtained. At the same times, optical microscope (OM) and transmission electron microscopy (TEM) were employed to investigate the microstructure characteristic. The experimental results showed that the recrystallization was more sensitive to temperature than holding time. The recrystallization process finished quickly above 1 050 ℃, and significantly prolonged below 1 025 ℃. Additionally, the dynamical model of static recrystallization follows the Avrami equation. The nucleating mechanism was characterized by bulging at grain boundary and merging of sub-grain.

Key words: Inconel 718 alloy; static recrystallization; nucleating mechanism

1 Introduction

Inconel 718 alloy, which is the typical precipitation hardened alloy, has a good usability and machining performance. Thus, this alloy is widely used in aerospace industry, petroleum chemical industry and other fields[1-4]. Undoubtedly, the excellent performance of Inconel 718 alloy is related to the reasonable microstructures[5-7], especially the appropriate grain size[8-10]. In order to gain a reasonable grain size for different operation conditions, it is necessary to control the heat treatment and processing parameters. So the study of recrystallization of Inconel 718 alloy is important for achieving the good properties. As the traditional rolling temperature range of Inconel 718 alloy sheet is always controlled between 1 000-1 100 ℃, the recrystallization behavior in this temperature range was studied in this article using two-stage interrupted compression method on Gleeble1500D thermal stimulator, and the dynamical model of static recrystallization was built. In addition, the recrystallization mechanism was analyzed by the observation of optical microscope (OM) and transmission electron microscopy (TEM).

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2 Experimental

The investigated alloy was prepared through a double melting process: vacuum induction melting (VIM) plus electro slag remelting (ESR). After homogenizing treatment, it was forged to rod with 40 mm in diameter. The chemical compositions are shown in Table 1. The cylindrical specimens for hot compression test with 8 mm in diameter and 15 mm in height were then cut from the forging bar. To study the recrystallization behavior of Inconel 718 alloy between 1 000-1 100 ℃, two-stage interrupted compression tests were performed by Gleeble 1500D simulator. Prior to the deformation, the samples were soaked at 1 180 ℃ for 5 min, then cooled to the deformation temperature. After a deformation up to a reduction of 20% at a strain rate of 1/s followed by holding for 1-3 000 s, the second deformation reduction of 20% was also applied as illustrated in Fig.1.

Table 1 The chemical compositions of the Inconel718 alloy /wt%

C	Cr	Mo	Al	Ti	Nb	Co	Cu	Fe	Mn	Si	B	P	S	Ni
0.024	17.99	3.12	0.55	0.99	5.19	0.024	0.0039	18.87	0.013	0.072	0.0026	0.002	0.002	Bal

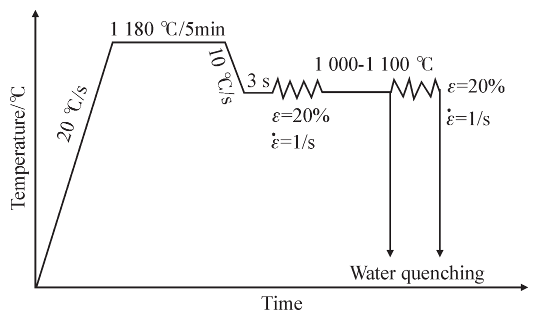

Fig. 1 Schematic diagram of two stage interrupted compression test

Recrystallization fraction (X_{SRX}) was gained by comparing the two flow stress values, and the 2% offset method was used by analyzing the experimental data[11,12]. Therefore, the recrystallization fraction (X_{SRX}) is defined in Eqs.(1). And a typically stress-strain curve of two-stage interrupted compression is shown in Fig.2.

$$X_{SRX}=\frac{\sigma_m-\sigma_{r,2\%}}{\sigma_m-\sigma_{0,2\%}}\times 100\% \tag{1}$$

where, σ_m is the maximum flow stress in the first curve, and $\sigma_{0,2\%}$ and $\sigma_{r,2\%}$ are the flow stress in the first and second curve at a plastic strain of 2%, respectively.

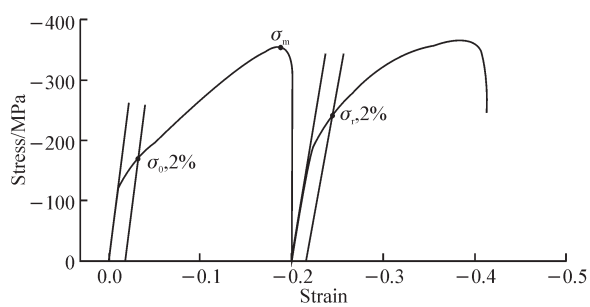

Fig.2 Stress-strain curve of two stage interrupted compression test used to determine the recrystallization fraction

To observe the recrystallization behavior of Inconel 718 alloy, specimens were water quenched to room temperature after holding for a certain time, after the first deformation up to a reduction of 20% at a true strain rate of 1/s as shown in Fig.1. Then, the compressed specimen was cut along with the axis direction, and the TEM samples were cut parallel to the profile. Specimens were cut from the center of the slice with 3 mm in diameter and reduced in methanol solution with 15vol% $HClO_4$ between −15 ℃ and −20 ℃, and the microstructure was observed by transmission electron microscopy (TEM).

3 Results and discussion

3.1 The stress-strain analysis

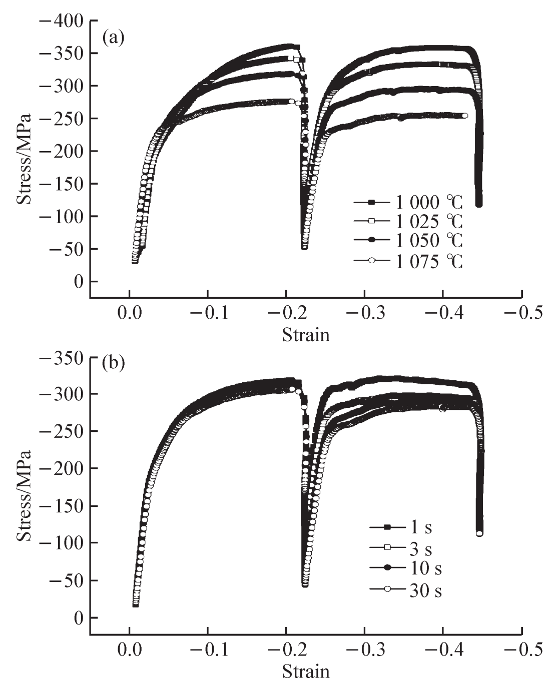

Fig. 3 Flow stress-to-strain curves of specimens (a) after holding for 10 s at different temperatures and (b) deformed at 1050℃ with different holding times

The stress-strain curves of some specimens after two-stage interrupted compression test are illustrated in Fig.3. The specimens were deformed at 1 000 ℃, 1 025 ℃, 1 050 ℃ and 1 075 ℃ followed by a hold for 10 s, the stress-strain curves are shown in Fig.3(a). It is clearly that the flow stress decreases significantly and the static recrystallization fraction increases with the rising of temperature within the same holding time. In addition, at the same deformation temperature, the flow stress decreases obviously and the static recrystallization fraction increases with the increasing of holding time, as shown in Fig.3(b). Moreover, with other deformation conditions, the variation rule of stress-strain curves and recrystallization fraction shows a similar trend.

3.2 Recrystallization fraction

Fig.4 shows that the recrystallization fraction of Inconel 718 alloy. It is changed with holding

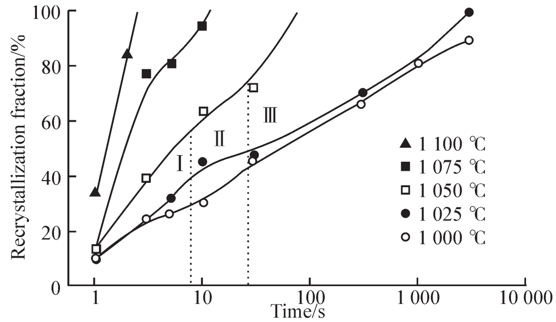

Fig.4 Recrystallization fraction curves of Inconel 718 alloy after deformation with the reduction of 20% at the temperatures ranging from 1000 to 1 100 ℃ and strain rate of 1/s

time during 1 000-1 100 ℃. The results show that the process of recrystallization is quickly finished at 1 050 ℃, 1 075 ℃ and 1 100 ℃, and the amount of time required to finish recrystallization significantly decreases with the increasing of temperature, which are 74 s, 12 s and 2.5 s at 1 050 ℃, 1 075 ℃ and 1 100 ℃, respectively. Even in some cases, the curve of recrystallization at different temperatures intersects with each other, such as the curve of recrystallization at 1 025 ℃ and 1 000 ℃, due to the sufficient nucleation energy at the beginning of recrystallization. And the amount of time required to finish recrystallization significantly increases, corresponding to 2 954 s and more than 3 000 s, respectively. Fig.5 shows the metallographic microstructure of specimens deformed at 1 025 ℃ and 1 050 ℃ followed by a hold for 2 954 s and 74 s, and the recrystallization fraction from the stress-stain curve conforms to optical microstructures. In addition, according to the change of slope of the recrystallization curves, some characteristic points could be identified in the recrystallozation curves at 1 075 ℃, 1 050 ℃ and 1 025 ℃. Before the appearance of transition point, the recrystallization proceeds quickly. It is considered that the driving force of recrystallization mainly comes from the distortion energy stored in the process of deformation, which corresponds to the initial stage (Ⅰ) of recrystallization curve at 1 050 ℃ in Fig.4. As the first characteristic point appears, the distortion energy is almost consumed by the recrystallized process. The mainly driving force is translated to heat transfer of the high temperature environment, which results in a certain induction period of recrystallization. Therefore, the slope of the curve is becoming more negative, as the second stage (Ⅱ) of recrystallization curve at 1 050 ℃ in Fig.4. When the nucleation of recrystallization finishes, the nucleus steps into the process of growth and finally the recrystallization finishes, as shown with the third stage (Ⅲ) of recrystallization curve at 1 050 ℃ in Fig.4. At the same time, the incubation period of recrystallization is not reflected in the recrystallization curve at 1 100 ℃, and which is short at 1 075 ℃, while significantly prolongs at 1 025 ℃, accompanying with the times of recrystallization in the range of 10 s to 1200 s, as shown in Fig.4.

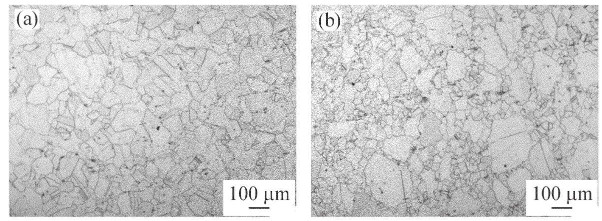

Fig.5 Optical microstructure of specimens after isothermal holding at (a) 1 025 ℃ for 2 945 s and (b) 1050 ℃ for 74 s

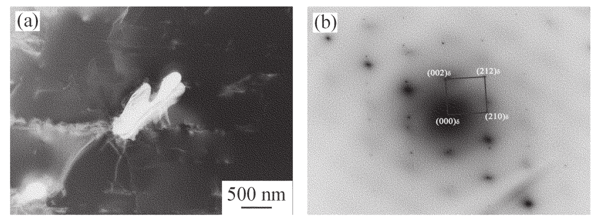

Fig.6 TEM images: (a) the precipitation of δ phase, and (b) its selected area diffraction pattern when the specimen deformed at 1 000 ℃ followed by a hold for 1 000 s

Additionally, δ phase could completely dissolve into austenitic matrix at 1 020 ℃ when the specimen is soaked at this temperature for a certain time[13]. Moreover, according to the research of Cai *et al*[13,14], the equilibrium concentration of δ phase in Inconel 718 alloy is about 0.6% (mass fraction) at 1 000 ℃, distributing along grain boundaries and presenting in particle. In this work, due to the solution treatment at 1 180 ℃ for 5 min, there is no δ phase precipitated in specimen before deformation. It is believed that the δ phase is precipitated when the specimen is holding at 1 000 ℃, and its morphology is shown in Fig.6. In fact, some activation energy is required for the proceeding of precipitation, recovery and recrystallization, so mutual influence and restriction relationship are existed among those three factors. That is, the process of recrystallization is inhibited by the precipitation of δ phase in some extent, which results in the decreasing of slope during 4 s to 10 s on the recrystallization curve of 1 000 ℃. However, the precipitation of δ phase could only affect the proceeding of recrystallization in some extent for its limited precipitation amount, so the recrystallization fraction increases with the increasing of holding-time.

3.3 Dynamical model of recrystallization

Many researches and experiments showed that the dynamical model of recrystallization for austenite usually accorded with the Avrami equation[15-17], which could be expressed as follows:

$$X_{\mathrm{SRX}} = 1 - \exp\left[-0.693 \left(\frac{t}{t_{0.5}} \right)^n \right] \tag{2}$$

where, X_{SRX} is the static recrystallization fraction, $t_{0.5}$ is corresponding to the time as 50% of recrystallization completed, and n is a constant. Eq.(3) could be obtained by taking logarithm on the both side of the Eqs.(2).

$$\ln \frac{\ln(1 - X_{\mathrm{SRX}})}{-0.693} = n \ln \frac{t}{t_{0.5}} + \ln c \tag{3}$$

where, c is a constant. Then, based on the experimental data in Fig.3, linear regression analysis was used to gain the value of n and R^2 (correlation coefficients) at different temperatures, and the results are shown in Table 2.

Table 2 The value of n and R^2 at different deformed temperatures

Temperature/℃	The value of n	The value of R^2
1 000	0.355	0.970
1 025	0.358	0.943
1 050	0.581	0.981
1 075	1.182	0.932
1 100	2.139	0.997

The data from Table 2 show that the dynamical model of recrystallization for Inconel 718 alloy could be depicted by Avrami equation effectively, and the susceptibility of the recrystallization to time is relatively low below 1 025 ℃. In this temperature range, the value of n remains basically invariable. However, the value of n raises quickly above 1 050 ℃. Within the temperature range of 1 050-1 100 ℃, the recrystallization fraction increases significantly with the increasing of time, as illustrated in Table 2. It also shows that the recrystallization could be completed quickly during 1 050-1 100 ℃, while requires a longer holding time during 1 000-1 025 ℃.

3.4 Recrystallization-nucleation mechanism

The recrystallization-nucleation mechanisms of metal materials are studied extensively[18-20], including classical nucleation theory, boundary nucleation, merging of sub-grain, particle stimulated nucleation, twin crystal nucleation, *etc*.

Fig.7 shows the microstructure of specimens with isothermal holding at 1 000 ℃ for 5 s and 1 050 ℃ for 10 s after the first deformation. Obviously, the bulge of grain boundary is in the shape of sawtooth, and there are a large number of fine recrystallization grains around grain boundaries, but those fine recrystallization grains do not appear in transgranular. By analyzing the microstructures, it could be concluded that the mode of nucleation of Inconel 718 alloy is mainly bulging at grain boundary in the process of high temperature deformation.

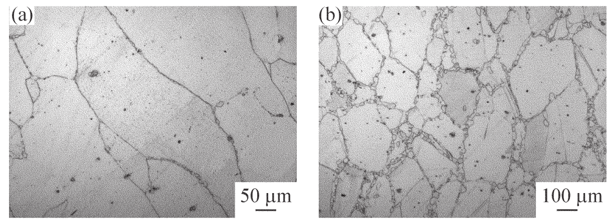

Fig.7 Optical micrographs of specimens after isothermal holding at (a) 1 000 ℃ for 5 s and (b) 1 050 ℃ for 10 s

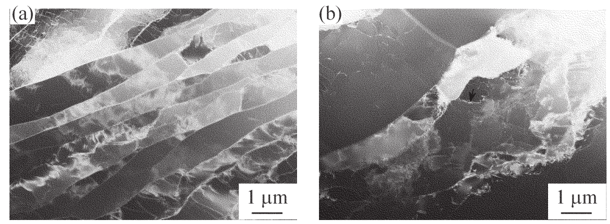

Fig.8 TEM images of Inconel 718 alloy held for 100 s at (a) 1 000 ℃ and (b) 1 025 ℃

When the Inconel 718 alloy is compressed at high temperatures, and various sizes of sub-grains are forming on the two sides of original high angle boundary. Fig. 8 shows the TEM microstructures with isothermal holding for 100 s at 1 000 ℃ and 1 025 ℃. Obviously, the dislocation pile-up and dislocation wall are formed due to the produced dislocation during deformation, which results in the formation of sub-grain, as illustrated in Fig.8(a). At the same times, the new grain, which is formed during the deformation and isothermal holding process, is shown in Fig.8(b) with arrows. In order to decrease the nucleation energy, the boundary is bulged to the side of high density dislocation. Because the driving force of recrystallization mainly comes from the decreasing of the free energy of matrix, the boundary of nucleus always shifts along with the opposite direction of the curvature centre, and the migration rate of grain boundary is inversely related to the radius of curvature.

Therefore, the recrystallization behavior of Inconel 718 alloy is mainly bulging at grain boundary in the process of high temperature deformation, accompanying with the merging of sub-grain.

4 Conclusions

a) The static recrystallization fraction of Inconel 718 alloy was obtained using two-stage interrupted compression method on Gleeble1500D thermal stimulator. And the rate of recrystallization increased significantly with the increasing of temperature. Recrystallization was completed quickly above 1 050 ℃, and prolonged obviously below 1 025 ℃.

b) The dynamical model of recrystallization of Inconel 718 alloy followed the Avrami equation, the time index almost presented in the same value level below 1 025 ℃, was about 0.355, and increased significantly above 1 050 ℃, which increased from 0.581 to 2.139 as the temperature increased from 1 050 ℃ to 1 100 ℃.

c) The recrystallization-nucleation mechanism of Inconel 718 alloy was mainly bulging at grain boundary, accompanying with the merging of sub-grain under the deformation of 20%.

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