Hot Deformation Behavior of 1Cr12Ni3Mo2VN Martensitic Stainless Steel

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Abstract: 1Cr12Ni3Mo2VN is a new type of martensitic stainless steel for the last-stage blades of large-capacity nuclear and thermal power turbines. The deformation behavior of this steel was studied by thermal compression experiments that performed on a Gleeble-3500 thermal simulator at a temperature range of 850°C to 1200°C and a strain rate of 0.01s⁻¹ to 20s⁻¹. When the deformation was performed at high temperature and low strain rate, a necklace type of microstructures was observed, the plastic deformation mechanism is grain boundary slip and migration, when at low temperature and lower strain rate, the slip bands were observed, the mechanism is intracrystalline slips, and when at strain rate of 20s⁻¹, twins were observed, the mechanism are slips and twins. The Arrhenius equation was applied to describe the constitutive equation of the flow stress. The accuracy of the equation was verified by using the experimental data and the correlation coefficient R² = 0.9786, and the equation can provide reasonable data for the design and numerical simulation of the forging process.

1 Introduction

1Cr12Ni3Mo2VN (French grade: E-Z12CNDV12, British grade: S/SJ2, Japan grades: KT5312AS6) martensitic stainless steel is a material commonly employed in manufacturing the last-stage blade which are the key components of nuclear power and thermal ultra-supercritical steam turbines. The service environments of the blades are very bad with high temperature, high pressure and humid, the requirements of the blades should be high corrosion resistance, high temperature creep performance, high fracture toughness, high fatigue strength and oxidation resistance [1-3], it is very important to investigate the hot deformation behavior of this steel for developing hot deformation processes.

At present, the research methods of hot deformation behavior are mainly through the thermal tensile or thermal compression test to obtain the stress-strain curves, and the Zener-Holloman parameter [4] was introduced into Arrhenius equation that describes the high temperature constitutive equation [22] as listed below:

\[ Z = \dot{\varepsilon} \exp \left( \frac{Q}{RT} \right) = A[\sinh(\alpha\sigma)]^n \]  \hspace{2cm} (1)

\[ \dot{\varepsilon} = AF(\sigma) \exp \left( -\frac{Q}{RT} \right) \] \hspace{2cm} (2)
where \( \dot{\varepsilon} \) is strain rate, \( Q \) is hot deformation activation energy, \( R \) is the gas constant with value 8.314 J/(mol-K). \( T \) is the absolute temperature, \( \sigma \) is flow stress, and \( A, n_1, \alpha, \beta, n \) are material constants with \( \alpha = \beta/\eta_1 \).

Many works [5-15] were done to establish the Arrhenius constitutive equations by linear fitting method that utilized the peak stresses of the thermal experiments, and the flow stresses were predicted by the equations. Li et al. [16] studied the hot deformation behavior of blades steel 10Cr12Ni3Mo2VN for ultra-supercritical steam turbine, a Levenberg-Marquardt algorithm was introduced to calculate the material constants, and the relationship between the critical strain and the \( Z \) parameter was given, the critical strain of the dynamic recrystallization was also studied by Cao et al [11], Laasraoui & Jonas [23] and Najafizadeh & Jonas [24]. Although the Arrhenius equation was accepted widely, the effect of strain on the stress dose not taken into account, and the predicted stresses were only fit to the peak stresses well. A strain compensation method [8, 13] was introduced to correct the predicted stresses, and the results were more accurate. The Sellars model were adopted by Huang et al [14] and Lin et al [15] to establish the constitutive equations of dynamic recovery and dynamic recrystallization respectively according to the critical strain.

1Cr12Ni3Mo2VN belongs to Cr-Ni-Mo-V martensitic heat-resistant stainless steel. Many studies on this steel were mainly focused on the control of the microstructure by heat treatment process [16-23]. Except Li [5], there are few reports on its thermal deformation behavior, and it is necessary to study its thermal deformation behavior. The purpose of this paper is to investigate the influence of deformation conditions on dynamic recrystallization, the plastic deformation mechanism and the flow stress constitutive equation and the strain compensation equations.

2 Experiment Method
The 1Cr12Ni3Mo2VN steel, which was in the annealing state and with chemical composition (wt%) of (bal.)Fe-0.144% C-0.816% Mn-11.47% Cr-1.53% Mo- 2.81% Ni-0.316% V-0.0323% N, was used in this study. The samples were cut into a cylindrical shape with a size of Ø10 x 15 mm. The experiments were performed on a Gleeble-3500 thermal simulator with a deformation temperature of 1200, 1150, 1100, 1050, 1000, 950, 900, 850°C and a strain rate of 0.01, 0.1, 1, 10, 20s\(^{-1}\). The samples were heated to 1200°C at a heating rate of 10°C/s and held for 3 minutes. After being completely austenitized, the samples were cooled to a deformation temperature at 10°C/s and held for 60s. Before compressing, two tantalum tablets were pasted the end of the samples, the compression was performed with a constant temperature and strain rate, and at the end the of compression, the height reduction was 60%. The samples were quenched to room temperature in water immediately after the compression finished. The strain, stress, temperature, stroke and other data were recorded, and the flow stress curves were drawn.

After the experiment, the specimens were cut into two parts along the middle axis, polished and eroded by the method introduced in paper [5], and the metallurgical microstructures were observed by DM ILM optical microscope.

3 Results and Discussions
3.1 Flow stress curves and metallurgical structure
The temperature of metal deformation has a great influence on the flow stress. Some flow stress-strain curves of 1Cr12Ni3Mo2VN obtained for different deformation conditions are shown in Figure 1. When the deformations are carried out at temperature higher than 1000°C and strain rate of 0.01s\(^{-1}\), the curves exhibited the typical characteristics of the dynamic recrystallization with a single peak value.
and a static stress. However the peak of the flow stress curve gradually disappears as the temperature decreases and the strain rate increases (Fig. 1). The fluctuation amplitude of the flow stress at strain rate of $1 \text{s}^{-1}$ (Fig 1b) is significantly larger than that at the strain rate of $20 \text{s}^{-1}$ (Fig 1c) and of $0.01 \text{s}^{-1}$ (Fig 1a). When the strain rate reaches $20 \text{s}^{-1}$, the front part of the flow stress curve fluctuates violently, and the curve is smooth after strain is greater than 0.2.

Fig. 1 Flow stress curves at different deformation conditions

The DRX microstructures at different temperatures and strain rate of $0.01 \text{s}^{-1}$ are shown in Figure 2. The microstructure is the elongated initial grains with very small recrystallization grains formed at the grain boundary at 850°C (Fig. 2a), with increasing the temperature, more and larger new grains formed at the initial grain boundary, and a necklace structure had formed (Fig. 2b & c). When the deformation temperature is higher than 1000°C, the initial structure disappeared, all the structure is instead of new recrystallization grains (Fig. 2d). With the deformation continues, the recrystallization grains are deformed to cause strain hardening, and the secondary recrystallization occurred (Fig. 2d, e, f), and a mixed crystal microstructure is formed. Because of the softening of secondary recrystallization, the flow stress curves are exhibited the fluctuations.

When the deformation temperature is higher than 1000°C with strain rate of 0.01s$^{-1}$, the governing softening mechanism is dynamic recrystallization, and the dynamic recovery is supplemented, and the flow stress curve exhibits typical dynamic recrystallization features.
(a) 850°C

(b) 900°C

(c) 950°C

(d) 1000°C

(e) 1100°C
Fig. 2 Optical photos of samples deformed at different temperatures and strain rate of 0.01s$^{-1}$

When the deformation temperature is lower than 950°C and the strain rate of 0.01s$^{-1}$, the dynamical recrystallization is partially completion, with the decreasing of temperature, the proportion of recrystallized microstructure is also decreasing, the effect of strain hardening is greater than the softening effect of recovery and recrystallization, the flow stress curve has no obvious peak and the stress has been increasing.

Fig. 3 Flow stress curves at different strain rates with constant temperature 1200°C

The flow stress-strain curves at different strain rates with constant temperature of 1200°C are shown in Figure 3. The higher the strain rate is, the greater the flow stress is, but the peak stress is less and less obvious, but the curve still fluctuates, that indicates the dynamic recrystallization occurs.

The plastic deformation mechanism of the polycrystalline metal at lower temperatures is intracrystalline slips and twins. The microstructures at different strain rates and the temperature of 850°C are shown in Figure 4. When the strain rate is lower than 1s$^{-1}$, only the slip bands are observed (Fig 4a, b), the slip bands in a same grain are parallel to each other, and terminates at the grain boundary, the slipping directions in different grains are also different. Because the slipping process is relatively gentle, the corresponding flow stress curve is relatively smooth; the twins are observed in the high strain rate of 20s$^{-1}$ (Fig. 4c), because of the twins often occurs suddenly, the flow stress curves exhibits sharply serrated fluctuations (Fig. 1c).
3.2 Plastic deformation mechanism

Fig. 4 Optical photos of samples deformed at different strain rates with temperature 850°C

The plastic deformation mechanism at high temperature is grain boundary slip and migration [25]. Due to the high temperature, only a small stress is applied, and the gap atoms and vacancies at the grain boundary migrate and plastic deformation occurs. The higher the temperature, the smaller the applied stress.

3.3 Constitutive equations of flow stress

The Arrhenius constitutive equation (2) are adopted to describe the flow stress-strain curves. The material constants can be estimated by fitting the equation (2) to the peak stress of flow stress curves, which first proposed by Sellars & McTegart [22]. Because the peak stress of the flow stress curves are not obvious, taking the stresses of ε is 0.1, 0.2, 0.3, 0.4, 0.5, 0.6 and 0.7 to replace the peak stress, the fitting results of the material constants are shown in Table 1.

| ε  | α     | n    | Q     | lnA  |
|----|-------|------|-------|------|
| 0.1| 0.0092| 6.2  | 446425| 37.57|
| 0.2| 0.007668| 5.965| 438389| 37.37|
| 0.3| 0.007618| 5.658| 434187| 36.79|
The relationship between the material constants ($\alpha$, $n$, $Q$, $A$) and strains are fitted by 4-order polynomial (3) (Wang et al, 2011; Wei et al, 2013), the fitting coefficients are shown in Table 2.

$$\alpha = A_0 + A_1 \varepsilon + A_2 \varepsilon^2 + A_3 \varepsilon^3 + A_4 \varepsilon^4$$
$$n = B_0 + B_1 \varepsilon + B_2 \varepsilon^2 + B_3 \varepsilon^3 + B_4 \varepsilon^4$$
$$Q = C_0 + C_1 \varepsilon + C_2 \varepsilon^2 + C_3 \varepsilon^3 + C_4 \varepsilon^4$$
$$\ln A = D_0 + D_1 \varepsilon + D_2 \varepsilon^2 + D_3 \varepsilon^3 + D_4 \varepsilon^4$$

| $\alpha$ | $n$ | $Q$ | $\ln A$ |
|---------|-----|-----|---------|
| $A_0$   | $B_0$ | $C_0$ | $D_0$ |
| 0.0134  | 6.1060 | 453972 | 36.35 |
| $A_1$   | $B_1$ | $C_1$ | $D_1$ |
| -0.0618 | 3.4426 | -77617.1 | 22.30 |
| $A_2$   | $B_2$ | $C_2$ | $D_2$ |
| 0.2261  | -30.508 | -10345.8 | -121.17 |
| $A_3$   | $B_3$ | $C_3$ | $D_3$ |
| -0.0337 | 56.052 | 151553 | 203.74 |
| $A_4$   | $B_4$ | $C_4$ | $D_4$ |
| 0.1761  | -31.932 | -82689.4 | -111.36 |

Fig 5 Relationship between experiment stress and predicted stress

Taking the experimental data for the horizontal axis, and taking the predicated data that calculated by equation (2, 3) as the vertical axis, the relationship between the two are shown in Figure 5. The data correlation judgment coefficient $R^2$ is 0.9786, the constitutive equation can provide a reliable data for forging process design and simulation.

4 Conclusions

Through the hot compression experiment of 1Cr12Ni3Mo2VN blade steel, the flow stress-strain curves and the microstructures of the samples after hot compression are analyzed. It can be concluded that:

Deformation at high temperatures and low strain rates, the flow stress-strain curves exhibits a typical dynamical recrystallization curve with a stress peak and static stress. With decreasing of temperature and increasing of strain rate, the peak of the flow stress-strain curve disappears, but the curve still
fluctuates to indicate the dynamical recrystallization. When the strain rate reaches 20s\(^{-1}\), the flow stress-strain curve fluctuates sharply at the beginning of the compression, and the fluctuation gradually disappears with the deformation continuing, the curves becomes a smooth curve until the strain great than 0.2. When the deformation temperature is constant, the flow stress increases with the increase of the strain rate, indicating that 1Cr12Ni3Mo2VN is a strain rate sensitive material.

Deformation at high temperatures and low strain rates, the plastic deformation mechanism of 1Cr12Ni3Mo2VN is grain boundary slip and diffusion. Deformation at low temperatures and low strain rates, the mechanism is intracrystalline slip, and deformation at low temperature and high strain rate, the mechanism is intracrystalline slip and twin, because the twin often occurs suddenly, the flow stress-strain curves exhibit sharply jagged fluctuations.

The Arrhenius constitutive equation (2) is used to describe the flow stress-strain curves, the material constants \((\alpha, n, Q, A)\) are estimated by fitting equation (2) to the experimental data of different strains. And the relationship between the parameters and the strains is fitted by polynomials (3). The constitutive equation is verified by experimental data, and the correlation coefficient \(R^2\) is 0.9786.

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