Research Article

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Hot deformation behavior and processing maps of 9Cr3W3Co oxide dispersion-strengthened steel

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Abstract: The hot deformation behavior of as-cast 9Cr3W3Co oxide dispersion-strengthened (ODS) steel was investigated by Gleeble 3500 facility in the temperature range of 900–1,150°C and at strain rates range of 0.01–10 s⁻¹. The constitutive equation and processing maps were established to describe this complex hot deformation process. The true stress–strain curves showed that the softening effects of dynamic recovery and dynamic recrystallization are stronger than the effect of work hardening with further increasing the temperature and strain. The optimal hot working parameters of 9Cr3W3Co-ODS steel are suggested to be $T = 1,050–1,100°C$ and $\dot{\varepsilon} = 0.03–0.3$ s⁻¹.

Keywords: heat-resistant steel, constitutive equations, processing maps, microstructure evolution

1 Introduction

The 9Cr3W3Co steel is well-known as one of the best materials with long service life under high pressure and temperature owing to their unique characteristics, such as high thermal conductivity, low thermal expansion coefficient, and so on [1,2]. In addition, steel with good high-temperature properties by adding oxide (e.g., $Y_2O_3$) is called oxide dispersion-strengthened (ODS) steels. To gain an excellent high-temperature property of 9Cr3W3Co-ODS heat-resistant steels, forming processes are continually updated and improved. It's generally known that microstructure, grain, and crack can be optimized by hot deformation. Accordingly, it's important to study the changes in microstructure and properties with different hot deformation parameters and then optimize the hot deformation process.

To date, large amounts of theoretical models are used to explain the thermal deformation behavior of the materials. For example, Prasad et al. [3] put forward the processing maps, which accurately described the hot workability of Ti-6242 alloy. Davenport et al. [4] employed a constitutive equation for modeling the forming processes of plain steel and microalloyed steel, which was distinguished from the traditional phenomenological constitutive models that were established by numerical statistics and neglected the physical meaning [5]. Based on the above models, Ebrahimi et al. [6] studied the microstructure evolution of heat-resistant steel (13% Cr) during hot deformation. Wang et al. [7] further investigated the critical $Z$ value, below which the dynamic recrystallization might occur. For 9Cr-ODS steel, Shao et al. have combined constitutive equation and processing maps to comprehensively describe the flow behaviors of 9Cr-ODS heat-resistant steel and obtained satisfactory results [8]. However, the abovementioned 9Cr-ODS was produced by mechanical alloying and hot isostatic pressing, which were different from the traditional production process (i.e., vacuum induction melting). There have been no reports on the study of hot deformation behaviors of 9Cr3W3Co-ODS steels with the traditional production process. Besides, the relevant constitutive models and processing maps are not yet clear.

In this article, the flow stress behavior of a 9Cr3W3Co-ODS steel were analyzed by isothermal compression tests with different processing parameters. The relevant
Theoretical models were carried to understand the hot deformation behaviors. Moreover, the microstructure evolution were also studied in detail under the different hot working parameters.

2 Experimental details

The designed alloy compositions are summarized in Table 1. Y₂O₃ was added at the end of vacuum induction melting. A nonaqueous solution electrolytic method [9] and field emission-scanning electron microscopy (FE-SEM) were used to extract and observe Y₂O₃ in the as-cast 9Cr3W3Co-ODS steel, as shown in Figure 1.

Isothermal compression tests were carried out in a Gleeble 3500 facility (Dynamic Systems Inc., Poestenkill, USA). Specimens were machined into cylinders by wire-electrode cutting with a diameter of 8 mm and a length of 15 mm. Based on the actual forging process (the beginning forging temperature is 1,150°C and the end forging temperature is 900°C), the experimental parameters of hot deformation are shown in Figure 2. Initially, all the samples were preheated to the complete austenitizing temperature (1,200°C) at a rate of 10°C/s and held for 15 min. The temperature was reduced from 1,200°C to the deformation temperature at a rate of 5°C/s and then compressed. Through the quenching, the high-temperature microstructures of the deformed samples were retained and prepared for observation. The deformed samples were polished, ultrasonically cleaned, and etched in a mixed solution of deionized water (100 mL), hydrochloric acid (50 mL), and FeCl₃ (5 g). The optical microscope (DM4000M, Leica, Germany) were used for investigating the evolutions of microstructure after deformation of samples.

3 Results and discussion

3.1 Flow stress curves

The flow stress curves of the studied 9Cr3W3Co-ODS steels under a range of hot compressive deformation parameters are given in Figure 3. The variational tendencies of curves are related to work hardening [10] and dynamic softening [11]. Moreover, the dynamic softening mechanisms contain dynamic recrystallization [12] and dynamic recovery [13]. During the infancy of hot deformation, the contribution of work hardening is stronger than that of dynamic softening, so that the stress increases rapidly. At this stage, dislocations begin to proliferate and move in the 9Cr3W3Co-ODS steels, which can effectively compensate for hot deformation [14]. As the strain increases, the curves tend to a decline or steady state, which depends on the competitive relationship between work hardening and dynamic softening. Clearly, the dynamic recrystallization and dynamic recovery play a major role. The softening effect of dynamic recovery increases gradually and the dynamic recrystallization grains begin to nucleate and grow with the increment of strain. In particular, both continuous and discontinuous dynamic recrystallization exist under the hot working process [15]. For the discontinuous dynamic recrystallization, it can be found at a large strain condition in the Figure 3(b). Because the dynamic recovery between the first-run and second-run recrystallization can’t counteract the work hardening, the stress value rises and the curve appears as a wave-like shape.

The values of peak stress and peak strain with different hot compressive deformation parameters are given in Table 2. The decrease in peak stress is followed by an increase in temperature. This is because that increasing temperature can accelerate atomic vibration and atomic diffusion [16], which are conducive to the dynamic recovery. Meanwhile, the higher temperature can also promote the movements of grain boundaries and then accelerate the dynamic recrystallization. By contrast, accompanied by an increase in the strain rate, the peak stress gradually increases. This is because that the faster strain rate can shorten the deformation time and restrain the effect of dynamic softening.

3.2 Constitutive analysis

The constitutive equation can well explain the flow behaviors and calculate the peak stress (σₚ) of 9Cr3W3Co-ODS

| Table 1: Composition of the as-cast 9Cr3W3Co-ODS steel (wt%) |
|-------------------|------|-----|-----|------|-----|-----|-----|-----|-----|-----|-----|-----|
| C                 | Si   | Mn  | Cr  | W   | Co  | Mo  | Ni  | V   | Nb  | N   | Y   | O   |
| 0.08              | 0.04 | 0.11| 9.6 | 3.0 | 2.8 | 0.12| 0.40| 0.21| 0.074| 0.015| 0.03 | 0.003| Bal. |
steels during steady hot deformation [17]. The strain rate ($\dot{\varepsilon}$) based on strain ($\varepsilon$) and other material constants can be calculated as follow [18,19]:

$$\dot{\varepsilon} = A \left[ \sinh(\alpha \sigma) \right]^n \exp \left[ \frac{-Q}{RT} \right]$$

(1)

where $Q$ are the activation energy (J/mol). And $\dot{\varepsilon}$, $R$, $T$, and $\sigma$ are corresponding to the strain rate (s$^{-1}$), universal gas constant [8.314 J (mol K)$^{-1}$], deformation temperature (K), and flow stress, respectively. Moreover, $A$, $\alpha$, $n$, and $\beta$ are the material parameters.

At low stress level ($a\sigma < 0.8$):

$$\dot{\varepsilon} = A_1 \sigma^n \exp \left[ -\frac{Q}{RT} \right] = B_1 \sigma^m$$

(2)

At high stress level ($a\sigma > 1.2$):

$$\dot{\varepsilon} = A_2 \exp(\beta \sigma) \exp \left[ -\frac{Q}{RT} \right] = B' \exp(\beta \sigma)$$

(3)

In equations (2)–(3), $n_i$, $B$, and $B'$ are the material parameters. $\alpha$ can be described as equation (4):

$$\alpha = \frac{\beta}{n_i}$$

(4)

Substituting the peak stress ($\sigma_p$) for flow stress ($\sigma$) in equations (1)–(3) and taking natural logarithm of both sides, we can easily find that

$$n_i = \frac{\partial \ln \dot{\varepsilon}}{\partial \ln \sigma_p}$$

(5)

$$\beta = \frac{\partial \ln \dot{\varepsilon}}{\partial \sigma_p}$$

(6)

Obviously, $n_i$ and $\beta$ can be linearly regressed to the relationship with $\ln \dot{\varepsilon}$-$\sigma_p$ (Figure 4(a)) and $\ln \dot{\varepsilon}$-$\sigma_p$ (Figure 4(b)), respectively. Taking logarithm of both sides of equation (1) and then taking partial derivative of 1/T, activation energy can easily find that

$$Q = Rn_2 b = R \left[ \frac{\partial \ln \dot{\varepsilon}}{\partial \ln (\sinh(\alpha \sigma_p))} \right] \left[ \frac{\partial \ln \left[ \sinh(\alpha \sigma_p) \right]}{\partial (1/T)} \right]$$

(7)

Using the same method as above, $Q$ can be obtained. The calculation results of hot deformation parameters of studied steels are summarized in Table 3.

In order to get the $A$ parameter, Zener and Hollomon et al. [20], in 1944, put forward one parameter named Zener–Hollomon parameter ($Z$), which was related to the hot compressive deformation parameters. $Z$ is defined as follows:

$$Z = \dot{\varepsilon} \exp \left( \frac{Q}{RT} \right)$$

(8)

$Z$ can also be derived from equations (1) and (8),
\[ Z = A \left[ \sinh(\alpha \sigma_p) \right]^n \]  

Taking logarithm of both sides of equation (9),
\[
\ln Z = \ln A + n \ln \left[ \sinh(\alpha \sigma_p) \right]
\]  

Therefore, \( \ln A \) and \( n \) are the intercept and slope of \( \ln Z - \ln[\sinh(\alpha \sigma_p)] \) [Figure 5(a)], respectively. Consequently, \( \ln A = 52.834, n = 4.507 \). In summary, the constitutive equation of the studied 9Cr3W3Co-ODS can be described as

### Table 2: Peak stress (\( \sigma_p \)) and peak strain (\( \varepsilon_p \)) of the samples with a series of hot compressive deformation parameters

| \( \dot{\varepsilon}/s^{-1} \) | \( T/°C \) | \( \sigma_p/MPa \) | \( \varepsilon_p \) | \( \dot{\varepsilon}/s^{-1} \) | \( T/°C \) | \( \sigma_p/MPa \) | \( \varepsilon_p \) |
|-----------------|----------|-----------------|----------|-----------------|----------|-----------------|----------|
| 0.01            | 900      | 122.071         | 0.039    | 1               | 900      | 197.754         | 0.054    |
|                 | 1,000    | 52.734          | 0.161    |                 | 1,000    | 147.949         | 0.064    |
|                 | 1,050    | 46.757          | 0.083    |                 | 1,050    | 115.234         | 0.088    |
|                 | 1,100    | 28.321          | 0.146    |                 | 1,100    | 64.453          | 0.576    |
|                 | 1,150    | 20.019          | 0.049    |                 | 1,150    | 46.387          | 0.415    |
| 0.1             | 900      | 137.695         | 0.024    | 10              | 900      | 223.145         | 0.161    |
|                 | 1,000    | 104.981         | 0.029    |                 | 1,000    | 165.039         | 0.093    |
|                 | 1,050    | 66.406          | 0.142    |                 | 1,050    | 121.094         | 0.2832   |
|                 | 1,100    | 38.574          | 0.171    |                 | 1,100    | 98.632          | 0.342    |
|                 | 1,150    | 26.856          | 0.176    |                 | 1,150    | 57.129          | 0.322    |
To describe the flow stress, we introduce the \( Z \) parameter in equation (11) as follows:

\[
\dot{\varepsilon} = 8.82 \times 10^{22} \times [\text{sinh}(0.0137\sigma_p)]^{4.507} \exp\left[-\frac{605248}{RT}\right]
\]  

Replacing the unknown constants with the material parameter, the flow stress equation of 9Cr3W3Co-ODS can be expressed as:

\[
\sigma = \frac{1}{a} \ln \left\{ \left(\frac{Z}{A}\right)^{1/n} + \left[\left(\frac{Z}{A}\right)^{2/n} + 1\right]^{1/2} \right\}
\]  

\[
\sigma = 72.99 \times \ln \left\{ \left(\frac{Z}{8.82E + 22}\right)^{1/4.507} + \left[\left(\frac{Z}{8.82E + 22}\right)^{2/4.507} + 1\right]^{1/2} \right\}
\]  

Table 3: Hot deformation parameters of the studied 9Cr3W3Co-ODS steels

| \( n_1 \) | \( n_2 \) | \( \beta \) | \( b \) | \( Q \) (kJ/mol) |
|---|---|---|---|---|
| 7.025 | 4.690 | 0.096 | 15.522 | 605.248 |

Peak stress \( \sigma_p \) can be calculated based on equation (13). Moreover, \( \sigma_p \) has important significance for the
hot deformation, which represents the maximum load. Figure 5(b) shows the relationship between the predicted peak stress and measured peak stress. All points are distributed near the oblique line with an angle of 45°. This oblique line means that the measured value is equal to the predicted value. It is obvious that the predictive value and the measured values are good identical ($R^2 = 0.96$). The result shows that the constitutive equation of 9Cr3W3Co-ODS steel is scientific and reliable.

### 3.3 Processing map

In order to further understand this complex process and gain the optimal deformation parameters, processing maps based on the data from flow stress curves have been established. The processing map theory is proposed by Raj [21], which is based on dynamic materials’ model. Power dissipation maps and instability maps contribute to the processing maps, and the total power can be given as:

$$P = \sigma \dot{\varepsilon} = G + J = \int_0^\varepsilon \sigma \dot{\varepsilon} \, d\varepsilon + \int_0^\sigma \dot{\varepsilon} d\sigma$$  \hspace{1cm} (14)

where $P$, $\sigma$, $\dot{\varepsilon}$, $G$, and $J$ are the total power, flow stress, strain rate, dissipated power in heat form, and reserved energy related to microstructure evolution (i.e., dynamic recrystallization and dynamic recovery), respectively.

The relationship between strain rate and stress can be expressed as:

$$\sigma = K\dot{\varepsilon}^m$$  \hspace{1cm} (15)

where $k$ represents the material parameter and $m$ represents the strain rate sensitivity coefficient.

$$m = \frac{\dot{\varepsilon} d\sigma}{d\dot{\varepsilon}} = \frac{df}{dG}$$  \hspace{1cm} (16)

When $m$ is 1, $J$ reaches a maximum value ($J_{\text{max}}$). The energy proportion of microstructure evolution can be reflected by power dissipation efficiency ($\eta$).

$$J_{\text{max}} = \frac{\sigma \dot{\varepsilon}}{2}$$  \hspace{1cm} (17)

$$\eta = \frac{J}{J_{\text{max}}} = \frac{2m}{m + 1}$$  \hspace{1cm} (18)

The power dissipation maps can be built by drawing the contour line of $\eta$ at a range of deformation temperatures and strain rates.

However, higher $\eta$ values do not mean the optimal hot deformation parameters. This is because the undesirable microstructure (e.g., wedge cracks) can also form in the high $\eta$ region [22]. So it’s a must that unsafe plastic flow should be further considered [23].

$$\frac{df}{d\dot{\varepsilon}} < \frac{J}{\dot{\varepsilon}}$$  \hspace{1cm} (19)

Substituting equation (16) into equation (19) can result in equation (20).

$$\xi(\dot{\varepsilon}) = \frac{\partial \ln \left( \frac{m}{m+1} \right)}{\partial \ln \dot{\varepsilon}} + m < 0$$  \hspace{1cm} (20)

where $\xi$ represents the instability parameter. Equation (20) is used to describe the criterion of microstructure.
Based on the true stree–strain curves, \( m \) can be obtained by cubic spline interpolation fitting at different hot compressive deformation parameters. The values of \( \eta \) and \( \xi \), in addition, can be calculated through equations (18) and (20).

The processing maps of the studied 9Cr3W3Co-ODS steels with the true strain of 0.4 and 0.6 are shown in Figure 6. The gray-shaded areas represent unstable regions. In Figure 6(a), the unstable region occurs for 900–1,080°C at strain rate range of 3.16–10 s\(^{-1}\). In Figure 6(b), the unstable region occurs for 900–1,150°C at strain rate range of 0.32–10 s\(^{-1}\). Compared with the processing maps of Figure 6(a), the unstable region becomes larger and expands into the high-temperature and low-strain rate region in Figure 6(b).

In the following, we focus the discussion on the processing map at the strain of 0.6 to analyze the effects of thermal deformation on microstructures of the studied 9Cr3W3Co-ODS steels. Figure 6(b) shows five different deformation regions (I–V): safe regions, which include positions I, II and III \((\xi > 0)\); unstable regions (gray-shaded area), which include positions IV and V.

Figure 7 shows the macro- and microstructure related to these five positions. At a relatively low hot working parameter, the optical microstructure characteristics of steady-state deformation are mainly the elongated structures, as shown in Figure 7(a). This is because austenite is seriously compressed along the axis direction and shows obvious directionality. The result shows that the evolution mechanism of hot deformation microstructure is dynamic recovery. With increasing hot working parameters, necklace structure begins to appear at the grain boundary, as shown in Figure 7(b). Necklace structure consists of fine and equiaxed new grains, which are distributed along austenite grain boundaries. Moreover, this is a signal of the beginning of dynamic recrystallization. Figure 7(c) shows the optical microstructure in position III compressed at 1,150°C with a strain rate of 1 s\(^{-1}\). In this position, the number of coarser DRX grains increases and some elongated grains can be observed. Moreover, the power dissipation efficiency is up to 40%, which indicates a higher microstructure evolution extent has taken place in the materials. Figure 7(d) and (e) show the typical crack types in the unstable region. At the low hot working temperature and high strain rate region \((\xi < 0)\), the fracture type is 45° shear fracture, which is caused by plastic instability [24]. Compared with Figure 7(d), the fracture type in Figure 7(e) changes to the oxidation crack at the highest strain rate and hot working temperature, which is caused by high temperature oxidation.

In conclusion, the hot processing maps of 9Cr3W3Co-ODS steel and experimentally observed macro- and microstructure have a great consistency. Moreover, flow instability occurs in the strain rate of 0.3–10 s\(^{-1}\). The high \( \eta \) occurs for 1,050–1,100°C at strain rates range of 0.03–0.3 s\(^{-1}\). Meanwhile, a large number of DRX grains appear in this region, which can significantly reduce the grain size and make the steel obtain high mechanical properties. So the optimal hot working parameters of the studied 9Cr3W3Co-ODS steels are suggested to be \( T = 1,050–1,100°C \) and \( \dot{\varepsilon} = 0.03–0.3 \) s\(^{-1}\).

![Figure 6](image-url): Processing maps of the studied 9Cr3W3Co-ODS steels at different strain: (a) 0.4; (b) 0.6. The gray-shaded area indicates unstable region. I–V represent five different deformation regions. Contour lines denote power dissipation efficiencies.
4 Conclusions

The hot deformation behavior of a 9Cr3W3Co-ODS was systematically investigated. The regions of the processing maps are analyzed in terms of the microstructural evolution under different hot working parameters and detailed conclusions are listed below:

1. The hot deformation process was affected by the work hardening and dynamic softening. The peak stress decreased as the hot deformation temperature increased or as the strain rate decreased.

2. The constitutive equation of the studied 9Cr3W3Co-ODS steel can be obtained as:

\[
\dot{\varepsilon} = 8.82 \times 10^{22} \times [\sinh(0.0137\sigma_p)]^{4.507} \exp\left[-\frac{605248}{RT}\right]
\]

3. Processing maps based of the true stress–strain curves can clearly describe this complex hot working process and help us obtain the optimal hot working parameters. The optimal hot working parameters of studied 9Cr3W3Co-ODS steels are suggested to be \( T = 1,050–1,100^\circ\text{C} \) and \( \dot{\varepsilon} = 0.03–0.3 \text{ s}^{-1} \).
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**Conflict of interest:** The authors declare no conflict of interest.

**Data availability statement:** The data used to support the findings of this study are included within the article.

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