Thermal Deformation Behavior of 10Cr12Ni3Mo2VN Blade Steel for Ultra-Supercritical Units (Postprint)

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Abstract

Compression experiments were conducted on 10Cr12Ni3Mo2VN steel using a Gleeble-1500 thermal simulator to investigate the hot deformation behavior under deformation temperatures of 850~1200 ℃ and strain rates of 0.01~10 s-1. The results show that the recrystallized grain size increases with increasing deformation temperature and decreasing strain rate. At a deformation temperature of 1200 ℃ and after 60% compression deformation, the recrystallized grains are equiaxed at higher strain rates, whereas mixed grains appear at lower strain rates. Hyperbolic sine constitutive equations for hot deformation were established using both the traditional linear fitting method and the Levenberg-Marquardt algorithm. The Levenberg-Marquardt algorithm enables simultaneous solution of all material parameters with simple procedure and reliable results, yielding a constitutive equation with higher predictive accuracy than the traditional method. By utilizing the work hardening rate-stress (-) curve and performing second-order differentiation, the critical strain was accurately determined, and relational equations between the critical strain, peak strain, and Zener-Hollomon parameter (Z parameter) were established.

Full Text

Hot Deformation Behavior of 10Cr12Ni3Mo2VN Blade Steel for Ultra-Supercritical Units

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Abstract

The hot deformation behavior of 10Cr12Ni3Mo2VN steel was investigated through compression tests using a Gleeble-1500 thermomechanical simulator at deformation temperatures ranging from 850 to 1200 °C and strain rates from 0.01 to 10 s ¹. The results demonstrate that recrystallized grain size increases with increasing deformation temperature and decreasing strain rate. At a deformation temperature of 1200 °C and 60% compression, equiaxed recrystallized grains form at higher strain rates, while mixed grain structures appear at lower strain rates. Hyperbolic sine constitutive equations for hot deformation were established using both traditional linear fitting methods and the Levenberg-Marquardt algorithm. The Levenberg-Marquardt approach enables simultaneous determination of all material parameters in a single step, offering a simpler procedure with reliable results and higher prediction accuracy compared to conventional methods.

Critical strain was accurately determined through secondary differentiation of work hardening rate-stress (-) curves, and relationships between critical strain, peak strain, and the Zener-Hollomon parameter (Z parameter) were established. These findings provide a theoretical basis for controlling the microstructure and properties of 10Cr12Ni3Mo2VN steel during hot working processes.

Keywords: 10Cr12Ni3Mo2VN steel; hot deformation; constitutive equation; critical strain

1. Introduction

10Cr12Ni3Mo2VN steel is a martensitic heat-resistant steel primarily used for manufacturing last-stage blades in ultra-supercritical power units. Due to its harsh service environment, this material demands strict microstructural property control [?, ?]. The steel is typically produced through forging processes, yet comprehensive studies on its hot deformation behavior remain scarce in the literature, necessitating thorough investigation.

Dynamic recrystallization occurs in metals under specific deformation conditions, and complete dynamic recrystallization is beneficial for achieving refined and uniform microstructural control. Numerous studies on dynamic recrystallization have been reported \cite{3~24}, with most employing hot deformation constitutive equations to investigate the influence of deformation parameters on recrystallization behavior. Several studies have also developed predictive models for critical strain of dynamic recrystallization \cite{4~11}. When establishing constitutive equations, some researchers have employed traditional methods involving partial differentiation and linear fitting \cite{5,9,10,12~16}, which cannot solve for all parameters simultaneously and require multiple steps. Cao et al. [?] proposed a minimum average difference optimization method that can obtain the complete constitutive equation at once, but the computational process is relatively complex. Therefore, developing a simple and effective method for solving hot deformation constitutive equations is of significant importance.

This work investigates the hot deformation behavior of 10Cr12Ni3Mo2VN steel through compression thermosimulation experiments. The study analyzes the effects of deformation parameters on microstructural evolution, establishes hyperbolic sine constitutive equations using the Levenberg-Marquardt algorithm, and develops relationships between critical strain, peak strain, and the Zener-Hollomon parameter. These results provide valuable guidance for microstructure and property control in 10Cr12Ni3Mo2VN steel.

2. Experimental Procedures

Test specimens were extracted from tempered 10Cr12Ni3Mo2VN forged material with the following chemical composition (mass fraction, %): C 0.11, Si 0.20, Mn 0.75, Cr 11.8, Ni 2.7, Mo 1.7, V 0.3, N 0.042, and Fe balance. Cylindrical specimens measuring 8 mm in diameter and 20 mm in length were machined for isothermal constant-strain-rate compression tests conducted on a Gleeble-1500 thermomechanical simulator.

The experimental procedure involved heating specimens at 10 °C/s to 1200 °C, holding for 3 minutes for austenitization, then cooling at 10 °C/s to deformation temperatures of 850, 900, 950, 1000, 1050, 1100, 1150, and 1200 °C, with an isothermal hold of 60 seconds at each temperature. Compression was then performed at constant strain rates of 0.01, 0.1, 1, and 10 s ^{1} to a maximum reduction of 60%, during which displacement-load curves were recorded. Immediately after compression, specimens were water-quenched to room temperature.

Following compression, specimens were sectioned along their longitudinal axis, polished, and electrolytically etched using an aqueous NaOH solution (40 g NaOH + 100 mL H O) to reveal grain boundaries. Microstructural examination was performed using an Axio Scope.A1 optical microscope (OM).

2.1 True Stress-True Strain Curves and Microstructural Morphology

The true stress-true strain curves of 10Cr12Ni3Mo2VN steel during hot deformation are presented in [Figure 1: see original paper]. Under all deformation conditions, the stress exhibits distinct peaks with increasing strain , indicating the occurrence of dynamic recrystallization [?]. The peak stress and corresponding peak strain increase with decreasing deformation temperature and increasing strain rate.

Microstructures of 10Cr12Ni3Mo2VN steel after 60% compression at a strain rate of 0.1 s 1 and various deformation temperatures are shown in [Figure 2: see original paper]. The volume fraction of dynamic recrystallization grains increases with deformation temperature, achieving complete dynamic recrystallization above 1150 °C. Additionally, recrystallized grain size increases with temperature due to enhanced thermal activation energy, which promotes elemental diffusion, dislocation slip and climb, and grain boundary migration [?].

[Figure 3: see original paper] and [Figure 4: see original paper] illustrate the microstructures after deformation at 1200 °C under different strain rates and reductions, respectively. At 1200 °C, complete dynamic recrystallization occurs after 60% compression at all strain rates (0.01, 0.1, 1, and 10 s 1), with average recrystallized grain size increasing as strain rate decreases. At higher strain rates (1 and 10 s 1), recrystallized grains are equiaxed ([Figure 3: see original paper]a and b), whereas mixed grain structures appear at lower strain rates (0.01 and 0.1 s 1) ([Figure 3: see original paper]c and d). As shown in [Figure 4: see original paper], complete dynamic recrystallization already occurs after 30% compression at 1200 °C with a strain rate of 0.1 s 1 ([Figure 4: see original paper]a), with further grain refinement occurring at larger deformations.

The formation of mixed grain structures at low strain rates and the phenomenon of grain refinement with increasing deformation can be attributed to the lower critical strain required for recrystallization under these conditions. Some regions undergo dynamic recrystallization first, and the newly formed grains have sufficient time to grow. As strain increases, grain growth and deformation proceed simultaneously. When the strain again reaches the critical value, a new cycle of dynamic recrystallization initiates, producing fine new recrystallized grains ([Figure 3: see original paper]d and [Figure 4: see original paper]c). However, this new recrystallization is only partial, resulting in irregular grain shapes and mixed grain structures.

2.2 Traditional Linear Fitting Method for Constitutive Equation

The relationship between deformation temperature, strain rate, and peak stress during hot deformation of metallic materials is expressed as [?, ?]:

$$\dot{\varepsilon} = A_1 \sigma_p^{n_1} \exp\left(-\frac{Q}{RT}\right) \quad \text{for } \alpha\sigma_p < 0.8 \tag{1}$$

$$\dot{\varepsilon} = A_2 \exp(\beta\sigma_p) \exp\left(-\frac{Q}{RT}\right) \quad \text{for } \alpha\sigma_p > 1.2 \tag{2}$$

$$\dot{\varepsilon} = A\left[\sinh(\alpha\sigma_p)\right]^n \exp\left(-\frac{Q}{RT}\right) \tag{3}$$

where $\dot{\varepsilon}$ is strain rate (s ¹); A, A_1, A_2, α, β, n, and n_1 are material constants with $\alpha = \beta/n_1$; σ_p is peak stress (MPa); Q is hot deformation activation energy (J/mol); T is deformation temperature (K); and R is the gas constant (8.3145 J/(mol·K)). Equation (1) applies at low peak stresses ($\alpha\sigma_p < 0.8$), equation (2) at high peak stresses ($\alpha\sigma_p > 1.2$), while the hyperbolic sine function in equation (3) is applicable over a wider range and better describes the relationship between deformation parameters and peak stress. The traditional solution method involves first solving for β and n_1, then obtaining an approximate value for α, followed by solving for remaining parameters.

Taking natural logarithms of equations (1) and (2) yields:

$$\ln\dot{\varepsilon} = \ln A_1 + n_1 \ln\sigma_p - \frac{Q}{RT} \tag{4}$$

$$\ln\dot{\varepsilon} = \ln A_2 + \beta\sigma_p - \frac{Q}{RT} \tag{5}$$

At constant deformation temperature, partial differentiation gives:

$$n_1 = \left[\frac{\partial \ln\dot{\varepsilon}}{\partial \ln\sigma_p}\right]_T \tag{6}$$

$$\beta = \left[\frac{\partial \ln\dot{\varepsilon}}{\partial\sigma_p}\right]_T \tag{7}$$

According to equations (6) and (7), n_1 and β can be obtained from the slopes of $\ln\dot{\varepsilon}$ vs. $\ln\sigma_p$ and $\ln\dot{\varepsilon}$ vs. σ_p plots, respectively. Since equation (1) is suitable for low peak stresses and equation (2) for high peak stresses, data at 1050-1200 °C were used to fit the $\ln\dot{\varepsilon}$-$\ln\sigma_p$ relationship, while data at 850-1000 °C were used for the $\ln\dot{\varepsilon}$-σ_p relationship, as shown in [Figure 5: see original paper]a and b. The average values obtained were $n_1 = 6.7348$ and $\beta = 0.03858$, giving $\alpha = \beta/n_1 = 0.00573$.

Taking the natural logarithm of equation (3) gives:

$$\ln\dot{\varepsilon} = \ln A + n\ln[\sinh(\alpha\sigma_p)] - \frac{Q}{RT} \tag{8}$$

Assuming constant deformation temperature and constant strain rate, respectively, partial differentiation yields:

$$n = \left[\frac{\partial \ln \dot{\varepsilon}}{\partial \ln[\sinh(\alpha \sigma_p)]} \right]_T \tag{9}$$

$$Q = R \left[\frac{\partial \ln[\sinh(\alpha \sigma_p)]}{\partial (1/T)} \right]_{\dot{\varepsilon}} \tag{10}$$

Based on equations (9) and (10), n and Q can be determined from the slopes of $\ln \dot{\varepsilon}$ vs. $\ln[\sinh(\alpha\sigma_p)]$ and $\ln[\sinh(\alpha\sigma_p)]$ vs. $1/T$ plots, respectively, as shown in [Figure 6: see original paper]. From the fitted slopes, the average values are $n = 5.799$ and $Q = 382.219$ kJ/mol.

The A value can be obtained from the $\ln \dot{\varepsilon} + Q/RT$ vs. $\ln[\sinh(\alpha\sigma_p)]$ relationship, shown in [Figure 7: see original paper]. From this relationship, $n = 5.773$, $\ln A = 34.31$, and $A = 7.95 \times 10^{14}$.

Introducing the Zener-Hollomon parameter (Z factor) [**?**, **?**, **?**]:

$$Z = \dot{\varepsilon} \exp\left(\frac{Q}{RT}\right) \tag{11}$$

The hyperbolic sine constitutive model for hot deformation becomes:

$$\dot{\varepsilon} = 7.95 \times 10^{14} \left[\sinh(0.00573\sigma_p)\right]^{5.7736} \exp\left(-\frac{382219}{RT}\right) \tag{12}$$

2.3 Novel Constitutive Equation Solution Method Based on Levenberg-Marquardt Algorithm

Currently reported methods for solving dynamic recrystallization hyperbolic sine constitutive equations primarily employ the traditional approach described above \cite{5,9,10,12~16}, where data are fitted using equations (1) and (2) to obtain parameters β and n_1 within limited ranges, followed by approximate determination of α in equation (3). However, the resulting α value is not a global solution for all data. Furthermore, parameters n and Q in the hyperbolic sine model are approximate averages from various deformation conditions rather than optimal solutions. The traditional method also requires multiple fitting steps, making the process complex and unable to determine all parameters simultaneously.

In this work, data analysis revealed that using the Levenberg-Marquardt algorithm in Matlab enables one-step fitting to obtain globally optimized solutions for all parameters in the dynamic recrystallization hyperbolic sine constitutive equation. The Levenberg-Marquardt algorithm is a well-developed and widely used method with mature models available in Matlab, allowing better consideration of relationships between global data and parameters through a simpler procedure.

Since the dynamic recrystallization hyperbolic sine constitutive equation is highly nonlinear, equation (3) was first transformed to improve fitting accuracy:

$$\sigma_p = \frac{1}{\alpha} \ln \left\{ (Z/A)^{1/n} + \left[(Z/A)^{2/n} + 1 \right]^{1/2} \right\} \tag{13}$$

where

$$Z = \dot{\varepsilon} \exp\left(\frac{Q}{RT}\right) \tag{14}$$

Experimental data were substituted into equation (14), and the Levenberg-Marquardt algorithm in Matlab was employed for fitting and solution.

The Levenberg-Marquardt algorithm requires appropriate initial values for convergence. Based on literature reports \cite{5,7,12~14}, the coefficients α, n, Q, and $\ln A$ for various steels are similar in magnitude and order. Computational results demonstrate that using α, n, Q, and $\ln A$ values from any steel reported in \cite{5,7,12~14} as initial values p_1, p_2, p_3, and p_4 yields excellent convergence, with final solutions showing negligible differences.

Using the Levenberg-Marquardt algorithm, simultaneous fitting yielded:

$$\alpha = 0.0059, \quad n = 5.96, \quad Q = 391938 \text{ kJ/mol}, \quad \ln A = 35.11 \tag{15}$$

The resulting hyperbolic sine constitutive model is:

$$\dot{\varepsilon} = 3.35 \times 10^{15} \left[\sinh(0.0059\sigma_p)\right]^{5.96} \exp\left(-\frac{391938}{RT}\right) \tag{16}$$

Parameters obtained from both traditional and Levenberg-Marquardt methods were substituted into equation (13) to calculate theoretical peak stresses under various deformation conditions. The relationship between measured and predicted σ_p values is shown in [Figure 8: see original paper]. Both methods exhibit high correlation coefficients ($R^2 > 0.99$) between predicted and measured values. Additionally, the average relative error δ was introduced [**?**]:

$$\delta = \frac{1}{N} \sum_{i=1}^{N} \left| \frac{\sigma_e - \sigma_t}{\sigma_e} \right| \times 100\% \tag{17}$$

where σ_e is the measured peak stress, σ_t is the predicted peak stress from the constitutive equation, and N is the number of experimental data points

($N = 32$). The average relative errors for traditional and Levenberg-Marquardt methods are 3.5% and 3.6%, respectively.

Furthermore, peak stresses at deformation temperatures of 1100 °C and 1200 °C with a strain rate of 0.01 s ¹ (66.9 MPa and 49.8 MPa, respectively) were excluded from the fitting process. The traditional method predictions for these conditions are 66.47 MPa and 45.42 MPa, while the Levenberg-Marquardt algorithm predictions are 67.82 MPa and 49.98 MPa, respectively.

These results demonstrate that both methods produce constitutive equations with high prediction accuracy. The coefficients obtained from the Levenberg-Marquardt algorithm are similar to those from the traditional method, validating the reliability of this approach. However, the Levenberg-Marquardt algorithm offers the advantage of simultaneously determining all hyperbolic sine constitutive equation coefficients in a single step, significantly simplifying the solution process.

2.4 Critical Strain Prediction Model

Determination of the critical strain ε_c for dynamic recrystallization is crucial for studying hot deformation. Current methods include metallographic observation and measurement of characteristic points on work hardening rate-stress ($\theta - \sigma$) curves ($\theta = d\sigma/d\varepsilon$) [**?**, **?**]. The metallographic method is labor-intensive, complex, and cannot precisely measure ε_c.

In this work, ε_c was determined from characteristic points on $\theta - \sigma$ curves, and a model relating ε_c and ε_p (strain at peak stress) to the Z parameter was established for predicting strain characteristics under various deformation conditions.

Since calculating work hardening rate θ involves differentiating the stress-strain curve, and experimental data consist of scattered points rather than smooth curves due to instrument noise, raw data were first fitted to obtain smooth curves. The $\theta - \sigma$ curves for 10Cr12Ni3Mo2VN steel at a strain rate of 1 s ¹ and various temperatures are shown in [Figure 9: see original paper]. As strain increases, work hardening rate gradually decreases due to dynamic recovery. When strain reaches the critical value, dynamic recrystallization occurs, enhancing softening and accelerating the decrease in work hardening rate, which appears as an inflection point on the $\theta - \sigma$ curve. Direct measurement of this inflection point is difficult; however, literature [**?**, **?**, **?**] indicates that when $d^2\theta/d\sigma^2 = 0$, it corresponds to the inflection point on the $\theta - \sigma$ curve, i.e., when $d\theta/d\sigma$ reaches an extremum, dynamic recrystallization initiates. [Figure 10: see original paper]a shows σ vs. $d\theta/d\sigma$ curves at a strain rate of 10 s ¹, enabling accurate determination of ε_c. [Figure 10: see original paper]b illustrates ε_c and ε_p at a strain rate of 10 s ¹.

For a given initial grain size, the relationship between ε_c, ε_p, and the Z parameter is expressed as [?, ?]:

$$\varepsilon_c = B_1 Z^{m_1} \tag{18}$$

$$\varepsilon_p = B_2 Z^{m_2} \tag{19}$$

where B_1, B_2, m_1, and m_2 are material constants. According to equations (18) and (19), these constants can be determined from $\ln \varepsilon_c$ vs. $\ln Z$ and $\ln \varepsilon_p$ vs. $\ln Z$ plots, as shown in [Figure 11: see original paper]. The resulting equations are:

$$\varepsilon_c = 0.0429 \ln Z - 3.241 \tag{20}$$

$$\varepsilon_p = 0.0375 \ln Z - 2.300 \tag{21}$$

These equations enable prediction of critical and peak strains under specific deformation conditions, providing a theoretical basis for developing hot working processes for 10Cr12Ni3Mo2VN steel.

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3. Conclusions

1. After hot deformation of 10Cr12Ni3Mo2VN steel at high temperature and high strain rates (1 and 10 s ^{1}), recrystallized grains are equiaxed. At low strain rates (0.01 and 0.1 s ^{1}), mixed grain structures form due to sufficient time for recrystallized grains to grow concurrently with deformation. When strain again reaches the critical value, new dynamic recrystallization occurs partially, resulting in mixed microstructures.

2. A method for establishing hot deformation hyperbolic sine constitutive equations using the Levenberg-Marquardt algorithm was proposed. Both traditional linear fitting and Levenberg-Marquardt methods produced constitutive equations with high prediction accuracy. Compared with traditional methods, the Levenberg-Marquardt algorithm simplifies the solution process by enabling simultaneous determination of all material parameters, yielding reliable results.

3. By utilizing work hardening rate-stress curves of 10Cr12Ni3Mo2VN steel and applying secondary differentiation, critical strain was accurately measured. Relationships between critical strain, peak strain, and the Zener-Hollomon parameter were established, allowing prediction of strain characteristics under specific deformation conditions and providing theoretical guidance for hot working process design.

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Note: Figure translations are in progress. See original paper for figures.