The metadynamic recrystallization behavior of ultrahigh-strength stainless steel: effects of precipitates and shear bands

Xiao-Hui Wang, Zhen-Bao Liu, Jian-Xiong Liang, Zhi-Yong Yang and Yue Qi

1 Institute for Special Steel Institute, Central Iron and Steel Research Institute, Beijing 100081, People’s Republic of China
2 Technical Center of Fushun Special Steel Shares Co. Ltd, Fushun 113006, People’s Republic of China

E-mail: liuzhenbao@nercast.com

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Abstract

The metadynamic recrystallization behavior of Cr-Co-Ni-Mo ultrahigh-strength martensitic stainless steel was studied in a double-pass isothermal compression test, and a metadynamic recrystallization kinetics model for softening was established. The results showed that the metadynamic recrystallization softening rate of the steel not only depended on the deformation temperature and strain rate but was also related to the dynamic precipitation and the local shear bands in the steel. When the deformation temperature was below 1050 °C, the dynamically precipitated M6C carbides pinned the grain boundaries and hindered metadynamic recrystallization. When the steel was deformed at a deformation temperature of 1000 ~ 1050 °C and a strain rate of 1.0 ~ 5.0 s⁻¹, a large number of local shear bands were generated. The local shear bands increased the number of nucleation sites for dynamic recrystallization and enhanced the softening rate of metadynamic recrystallization.

1. Introduction

Ultrahigh-strength stainless steel exhibits excellent mechanical properties and corrosion resistance and is therefore widely used in aviation, aerospace and other high-tech fields for such applications as aircraft landing gear, slat tracks and other key bearing components [1–3]. The manufacturing process of crucial aviation components usually includes multiple forging and heat-treatment steps. Forging guarantees that ultrahigh-strength stainless steel parts will obtain fine, uniform microstructures, thereby giving them excellent mechanical properties. During the forging process, the microstructure of steel usually undergoes dynamic recrystallization or dynamic recovery. Static or metadynamic recrystallization usually occurs between the two forging deformation steps or during the holding process after forging deformation. When the applied strain just exceeds the critical strain of dynamic recrystallization, metadynamic recrystallization will occur during the subsequent holding process. During this process, the dynamic recrystallization nuclei undergo continuous growth [4, 5]. The metadynamic recrystallization process will then significantly refine the deformed grain structure and thus affect the mechanical properties of the material. Therefore, studies on the metadynamic recrystallization behavior of ultrahigh-strength stainless steel have valuable applications.

The double-pass isothermal compression test is one of the most widely used methods for studying the dynamic softening mechanism and microstructure evolution of metadynamic recrystallization. For example, double-pass compression tests have been used to study the metadynamic recrystallization behavior of SA508-III steel [6], AISI 304L steel [7], 300M steel [8], nickel-based alloys [9] and magnesium alloys [10], as well as to establish a metadynamic recrystallization kinetics model. The above studies have shown that the dynamic softening mechanism and microstructure evolution of metadynamic recrystallization are very complicated. Paggi et al [7] studied the metadynamic recrystallization of AISI 304L and found that the grain growth behavior was mainly driven by strain-induced boundary migration. Zhao et al [8] studied the microstructure evolution mechanism of 300M steel via in situ observation methods. The main metadynamic recrystallization mechanisms of 300M steel were strain-induced boundary migration and curvature-driven grain boundary migration. Beladi et al [11] studied the metadynamic recrystallization mechanism of an Ni-30% Fe alloy. It was believed that the
metadynamic recrystallization softening process consisted mainly of the rapid growth of nuclei, the migration of grain boundaries and the combination of subgrain boundaries; in addition, the precipitated phase played a key role in metadynamic recrystallization. Pereloma et al. observed second-phase-induced metadynamic recrystallization nucleation in a Ni-30%Fe-Nb-C alloy. In summary, different alloys have different metadynamic recrystallization softening mechanisms. The studied steel in this work is an ultrahigh-strength martensitic stainless steel with an alloy content of more than 30 wt.% and exhibiting an ultrahigh strength above 1900 MPa, excellent fracture toughness and great corrosion resistance. However, because of the high content of alloying elements, e.g., Cr, Ni, Co and Mo, steel displays great high-temperature resistance, poor thermoplasticity and an obvious tendency toward dynamic precipitation during hot deformation. In the actual hot forging process, the strain and temperature fields are not uniform, which inevitably results in areas of incomplete recrystallization that affect the uniformity and stability of the material structure and properties. The metadynamic recrystallization in the subsequent heat-treatment process can further refine a coarse deformed structure that has not undergone dynamic recrystallization. Thus, metadynamic recrystallization plays an important role in obtaining a fine, uniform microstructure. In this study, the metadynamic recrystallization behavior of a novel ultrahigh-strength stainless steel is studied via a double-pass hot compression experiment, the metadynamic recrystallization kinetics equation is established, and the effects of the deformation parameters (strain rate and deformation temperature) and microstructure evolution on the metadynamic recrystallization behavior are discussed.

2. Experimental method

The composition of the stainless steel was 0.14 wt.% C, 13.0 wt.% Cr, 14.0 wt.% Co, 5.0 wt.% Mo, 2.0 wt.% Ni, 1.21 wt.% W, and 0.32 wt.% V, and the double vacuum smelting process (vacuum induction melting + vacuum arc remelting) was used. Steel rods with a diameter of 300 mm were forged after homogenization annealing at 1250 °C for 24 h. The double-pass compression test was then conducted using a Gleeble 3800 thermal simulator. Cylindrical specimens with diameters of 8 mm and 15 mm in height were used. A schematic diagram of the double-pass isothermal compression test is shown in figure 1. To ensure complete austenitization and a uniform grain structure before compression, the specimen was first heated to 1150 °C at a heating rate of 10 °C s⁻¹ and was held at 1150 °C for 5 min. Then the temperature was lowered to the deformation temperature at a cooling rate of 10 °C s⁻¹. The deformation temperatures of the first pass of each trial were 1000 °C, 1050 °C, 1100 °C and 1150 °C, and the strain rates were 0.1 s⁻¹, 1.0 s⁻¹ and 5.0 s⁻¹, respectively. The specimen was unloaded when the true strain reached 0.4. To study the deformed microstructure of the specimen after the first pass, a portion of the specimen was immediately water-quenched; the remainder was kept at the same deformation temperature for 1 s, 5 s, 10 s, 20 s, 50 s or 100 s, followed by second-pass deformation. The deformation temperature and the strain rate of the second pass were the same as those of the first pass, and the specimen was water-quenched immediately after the deformation. The hot compressed specimen was cut along the compression direction. After grinding and polishing, the specimen was corroded in a potassium permanganate-sulfuric acid solution (5 g KMnO₄ + 10 ml H₂SO₄ + 90 ml H₂O) for 24 h for metallographic observation. The evolution behavior of the metadynamically recrystallized grain structure was then studied using a ZEISS-40MA metallurgical microscope. The precipitated phase was characterized on a Tecnai G2 F30 S-TWIN transmission electron microscope.
TEM. The TEM sample was first mechanically polished to a thickness of less than 40 μm and then polished by an electrolytic double spray in a perchloric acid-alcohol solution (8% HClO$_4$ + 92% C$_2$H$_5$OH). The test temperature was −20 °C.

3. Experimental results

3.1. Stress-strain curve

To study the metadynamic recrystallization behavior of ultrahigh-strength stainless steel, a double-pass isothermal compression test was performed at strain rates of 0.1–5.0 s$^{-1}$, deformation temperatures of 1000–1150 °C and a true strain of 0.4. The stress-strain curve is shown in figure 2. Figure 2 (a) shows the stress-strain curves at a deformation temperature of 1100 °C, a strain rate of 0.1 s$^{-1}$ and different interpass times. The figure indicates that when the interpass time was in the range of 0–50 s, the yield stress of the second pass gradually decreased as the interpass time increased. When the interpass time was further increased from 50 s to 100 s, the yield stress almost did not change with the interpass time, indicating that complete metadynamic recrystallization softening had occurred in this time interval. There were two key factors that caused the second-pass yield stress to decrease with the interpass time. First, as the interpass time increased the metadynamic recrystallization fraction increased, and the accumulated dislocation density of the first-pass deformation decreased, resulting in a decreased yield stress of the second pass. Second, as the interpass time increased, the metadynamically recrystallized grain size increased, also resulting in a decrease in the yield stress. Figures 2 (b) and (c) show the effects of the strain rate and the deformation temperature on the flow stress. The flow stress gradually increased with an increase in the strain rate and with a decrease in the deformation temperature.

3.2. Metadynamic recrystallization softening fraction

The softening fraction was determined by the 0.2% offset stress method as follows [4, 9]:

$$F = \frac{\sigma_m - \sigma_2}{\sigma_m - \sigma_1}$$

(1)
where $\sigma_m$ (MPa) is the flow stress at the end point in the first stage of compression and $\sigma_1$ and $\sigma_2$ (MPa) are the offset yield strengths of the first- and second-pass deformations, respectively. In general, metadynamic recrystallization occurs when the softening fraction exceeds 0.2; thus, the metadynamic recrystallization softening fraction ($X_{mdrx}$) can be determined from the softening data using the following equation [4, 9]:

$$X_{mdrx} = \frac{F - 0.2}{0.8}$$

Figures 3 (a) and (b) show the metadynamic recrystallization softening fraction of the test steel at strain rates of 0.1 s$^{-1}$ and 1.0 s$^{-1}$ and deformation temperatures of 1000°C, 1050°C, 1100°C and 1150°C. At the same deformation temperature, the metadynamic recrystallization softening fraction increased as the interpass time increased. At the same interpass time, the metadynamic recrystallization softening fraction increased as the deformation temperature increased because the higher the deformation temperature was, the stronger the grain boundary migration and the faster the growth of the dynamic recrystallization nuclei, thereby resulting in a greater metadynamic recrystallization softening fraction under the same interpass time. It is worth noting that the softening fraction at 1000°C was significantly lower than those at 1050°C, 1100°C and 1150°C. It was inferred that there were other factors affecting the metadynamic recrystallization softening fraction in addition to the deformation temperature. Figures 3 (c) and (d) show the metadynamic recrystallization softening fraction of the test steel at deformation temperatures of 1000°C and 1100°C and strain rates of 0.1 s$^{-1}$, 1.0 s$^{-1}$ and 5.0 s$^{-1}$. At the same deformation temperature, the softening fraction of the steel increased as the strain rate increased because a higher dislocation density could accumulate at a higher strain rate in the same time period. This is mainly manifested in two aspects: on one hand, steel that deformed at a higher strain rate produced more dislocations in the same time period; on the other hand, the high strain rate reduced the time available for dislocation movement (dislocation annihilation or rearrangement), making it difficult for dynamic recovery to occur. The above factors led to a higher driving force for metadynamic recrystallization at higher strain rates.

### 3.3. Observation of the microstructure

To study the microstructure evolution during the metadynamic recrystallization process, the microstructure was characterized after the first-pass deformation under different deformation conditions (see figure 4). At 950
At 950 °C, the specimen exhibited a typical elongated, non-recrystallized structure after the first-pass compression, accompanied by a large number of local shear bands (see figures 4 (a) and (b)). When the deformation temperature was increased to 1000 °C (figures 4 (c) and (d)), a large number of dynamic recrystallization nuclei appeared at the prior austenite grain boundaries, and the number of local shear bands decreased. When the

Figure 4. Morphology of prior austenite grains and local shear bands of steel (center area of the specimens) under different deformation temperatures (T), strain rates (\(\dot{\varepsilon}\)) and true strain (\(\varepsilon\)).
deformation temperature was further increased to 1100 °C, the number of local shear bands was significantly reduced under a strain rate of 5.0 s⁻¹ (see figure 4(e)), and there were almost no local shear bands under a strain rate of 0.01 s⁻¹ (see figure 4(f)). It is worth noting that both the portion and the average grain size of recrystallized grains increased with a decrease in the strain rate (figures 4(e) and (f)), which is within our expectation since a higher strain rate leads to the plugging of high-density dislocations at grain boundaries to form dislocation cell/network structures, further resulting in the inhibition of nucleation/growth of dynamic recrystallization. Upon further increasing the deformation temperature from 1100 °C to 1150 °C, no local shear bands were observed within the strain rate range of 0.01∼5.0 s⁻¹ (figures 4(g) and (h)). Therefore, ultrahigh-strength stainless steel is prone to local plastic instability (such as local shear bands) under a low temperature and a high strain rate when it undergoes hot compression deformation.

According to our previous results with this steel [13], when the deformation temperature is below 1050 °C, M₆C carbides will dynamically precipitate in the steel. Therefore, the first-pass compression specimen was characterized by TEM (see figure 5). The results confirmed the existence of dynamically precipitated M₆C carbides in the steel after the first-pass compression.

4. Discussion

The fraction of metadynamic recrystallization is generally predicted using the Avrami equation as follows:

\[
X_{mdrx} = 1 - \exp \left[ -0.693 \left( \frac{t}{t_{0.5}} \right)^n \right]
\]

where \(n\) is the material constant, \(X_{mdrx}\) is the metadynamic recrystallization softening fraction, \(t\) is the interpass time, and \(t_{0.5}\) is the time corresponding to a metadynamic recrystallization volume fraction of 50%, which is determined by both the deformation temperature and the strain rate and can be calculated using the following equation:

\[
t_{0.5} = A\dot{\varepsilon}^p \exp \left( \frac{Q_m}{RT} \right)
\]

where \(A, p, q\) are the material constants, \(\dot{\varepsilon}\) is the strain rate (s⁻¹), \(T\) is the deformation temperature (K), and \(Q_m\) and \(R\) are known as the metadynamic recrystallization activation energy (kJ·mol⁻¹) and gas constant (kJ·mol⁻¹·K⁻¹), respectively. Taking the logarithm of both ends of equation (3) gives

\[
\ln \left( \ln \left( \frac{1}{1 - X_{mdrx}} \right) \right) = \ln 0.693 + n \ln t - n \ln t_{0.5}
\]

Via the linear regression method, the average values of \(n\) can be determined by the slope of the \(\ln t\) versus \(\ln (\ln (\frac{1}{1 - X_{mdrx}}))\) plots, which are shown in figure 6. Then, the mean value of \(n\) was obtained as 0.813. Taking the logarithm of both ends of equation (4) gives
The values of $t_{0.5}$ can be identified from the relationship between $X_m$ and the corresponding interpass time. The plots of $\ln t_{0.5} - \ln \varepsilon$ and $\ln t_{0.5} - 1/T$ can then be developed by substituting $t_{0.5}$, $\varepsilon$ and $T$ into equation (5), which are shown in figure 7 and figure 8. Finally, the values of $Q_{m}$, $p$ and $A$ can be estimated as 302.09 KJ/mol, $-0.241$ and $1.03 \times 10^{-11}$, respectively. Equations (3) and (4) can also be represented as follows:

$$
\ln t_{0.5} = \ln A + p\ln \varepsilon + \frac{Q_m}{RT}
$$

(6)

The metadynamic recrystallization softening fractions of the test steel at strain rates of 0.1 s$^{-1}$ and 5.0 s$^{-1}$ were calculated according to the metadynamic recrystallization kinetics equation. The results are shown in the curves of figures 9 (a) and (b) and compared with the measured softening fraction (scatter plot) under the same deformation conditions. At a deformation temperature of 1000 °C, the measured metadynamic recrystallization softening fraction gradually became smaller than the calculated value as the interpass time increased. At a deformation temperature of 1050 °C, the measured metadynamic recrystallization softening fraction gradually became larger than the calculated value. At higher strain rates (1.0 s$^{-1}$ and 5.0 s$^{-1}$), the measured value of the softening fraction was significantly greater than the calculated value. At a deformation temperature of 1100 °C, the measured metadynamic recrystallization softening fraction was equivalent to the calculated value. To investigate the above phenomenon, the microstructure of the specimen...
after the first-pass deformation and an interpass time of 10 s were characterized (figures 10 (a) and (b)). The dynamically recrystallized grains nucleated not only at the previous deformed austenite grain boundaries (figure 10 (a) zone A and figure 10 (b) zone C) but also at the local shear bands (figure 10 (a) zone B and figure 10 (b) zone D). In other words, the local shear bands generated via hot deformation are the preferred nucleation sites for dynamic recrystallization and can increase the metadynamic recrystallization softening rate [14–16].

The same phenomenon was also observed in the formation of deformation-induced martensite during cold rolling of AISI 316 stainless steel. The research of Naghizadeh and Mirzadeh suggested that as nucleation sites, the presence of isolated shear bands, shear band/grain boundary intersections and shear band intersections promotes the formation of the martensite phase [17]. The morphology of prior austenite grains of specimens figures 10 (a) and (b) after metadynamic recrystallization are shown in figures 10 (c) and (d). The prior austenite grains structure is typical fine equiaxed grain, but bimodal structure exists, however, compared with deformed microstructure, the grain structure is more uniform and fine after metadynamic recrystallization. Thus, we believe metadynamic recrystallization plays an important role in obtaining a fine, uniform microstructure.

Figures 10 (e) and (f) show that the metadynamic recrystallization softening mechanisms of ultrahigh-strength stainless steel are different in the low temperature/high strain rate regime and the high temperature/low strain rate regime. As shown in figure 10 (e1-e4), the metadynamic recrystallization softening in the high temperature/low strain rate regime (deformation temperature: 1100–1150 °C, strain rate: 0.1–1.0 s⁻¹) mainly depends on the growth of dynamic recrystallization nuclei at the prior austenite grain boundaries. However, in the low temperature/high strain rate regime (deformation temperature range: 1000–1100 °C, strain rate range: 1.0–5.0 s⁻¹), the metadynamic recrystallization softening depends not only on the growth of the dynamic recrystallization nuclei at the prior austenite grain boundaries but also on the dynamic recrystallization nuclei at
the local shear bands (figure 10 (f1-f4)). At a deformation temperature of 1000 °C, the dynamically precipitated M6C carbides (figure 5) pinned and hindered the metadynamic grain boundary migration at both the prior austenite grain boundaries and the local shear bands. Thus, the growth of the dynamic recrystallization nuclei at both the prior austenite grain boundaries and the local shear bands was hindered by the pinning effect of the precipitated phase, resulting in a lower metadynamic recrystallization softening rate. The measured value of the softening fraction was thus lower than the calculated value, as shown in the black curves and scatter plot in figures 9 (a) and (b). At a deformation temperature of 1050 °C, no dynamically precipitated M6C carbides were present in the steel. Metadynamic recrystallization was then dominated by local shear bands, which increased the metadynamic recrystallization softening rate. At a high strain rate (5.0 s\(^{-1}\)), the number of local shear bands was large, so the measured softening fraction was significantly greater than the calculated value (see the red curves and scatter plot in figures 9 (a) and (b)). At a deformation temperature of 1100 °C, no M6C carbide was
present in the steel, and the number of local shear bands was negligible (see figures 4 (e) and (f)). Without the effect of both M₆C carbides and local shear bands, the measured value and calculated value of the metadynamic softening fraction were similar (see the blue curves and scatter plot in figures 9 (a) and (b)).

5. Conclusions

On one hand, a high alloy content, e.g., Cr, Ni, Co and Mo, gives the ultrahigh-strength martensitic stainless steel excellent comprehensive mechanical properties and high corrosion resistance; on the other hand, it leads to great high-temperature resistance, poor thermoplasticity and an obvious tendency toward dynamic recrystallization behavior of ultrahigh-strength stainless steel was studied through double-pass isothermal compression tests at deformation temperatures of 1000~1150 °C and strain rates of 0.1~5.0 s⁻¹. The metadynamic recrystallization kinetics equation was then determined. The influences of M₆C carbides and local shear bands on metadynamic recrystallization softening were investigated through microstructure analysis. The following conclusions were drawn:

1) The apparent activation energy \(Q_{\text{am}}\) of metadynamic recrystallization was calculated to be 302.09 KJ/mol. The metadynamic recrystallization kinetics equation of the ultrahigh-strength stainless steel was determined as follows:

\[
X_m = 1 - \exp \left[ -0.693 \left( \frac{t}{t_{0.5}} \right)^{0.813} \right]
\]

\[
t_{0.5} = 1.03 \times 10^{-12} \exp \left( \frac{302090}{RT} \right)
\]

2) When the deformation temperature was below 1050 °C, M₆C carbides dynamically precipitated. The pinning effect of the carbides on the grain boundaries caused the metadynamic recrystallization softening rate to decrease.

3) When hot compression deformation was performed at deformation temperatures of 1000~1050 °C and strain rates of 1.0~5.0 s⁻¹, there were a large number of local shear bands in the steel. The number of local shear bands gradually decreased as the deformation temperature increased and the strain rate decreased. When the deformation temperature was above 1100 °C, almost no local shear bands formed during hot compression deformation. Dynamic recrystallization nuclei were then formed at the previous austenite grain boundaries and local shear bands. The local shear bands increased the number of nucleation sites available for dynamic recrystallization, enhancing the softening rate of metadynamic recrystallization.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

ORCID iDs

Xiao-Hui Wang 🐦 https://orcid.org/0000-0002-1560-5710

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