Kinetics of Metadynamic Recrystallization in Microalloyed Hypereutectoid Steels

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The isothermal kinetics of the recrystallization processes of vanadium microalloyed high carbon steels has been measured and modelled. The overall softening data were obtained by double hit compression tests performed at temperatures between 900 to 1050°C, strain rates of 0.01 to 1 s⁻¹, and inter-pass times of 0.1 to 30 s. The recrystallization behavior above and below and the critical strain for dynamic recrystallization was investigated. The results show that there is a transition strain region between where both static and metadynamic recrystallization take place during the inter-pass time. The results also revealed that V and Si have a strong solute drag effect, on the kinetics of metadynamic recrystallization. A kinetic model is proposed which takes the V and Si concentrations into account.

KEY WORDS: metadynamic recrystallization; transition strain; critical strain; microalloyed hypereutectoid steels; vanadium; silicon; high carbon steels.

1. Introduction

It is well known¹–⁵) that there are three different regions of post-deformation softening behavior, depending on the applied strain. Less than 10% strain leads to recovery alone. Between 10% and the critical strain for dynamic recrystallization, isothermal annealing proceeds in two stages; static recovery followed by classical static recrystallization. If the critical strain for dynamic recrystallization is exceeded, dynamically recrystallized nuclei form, and continue to grow when the specimen is unloaded. This process is called metadynamic recrystallization and does not involve an incubation time, since recrystallized nuclei are already present upon termination of the deformation.

The kinetics of metadynamic recrystallization are relatively rapid and it is not unusual for completion to occur during quenching after deformation.⁶) Nevertheless, the quantitative determination of the kinetics is necessary for accurate control of the recrystallization process, especially when considering rolling schedules with very short interpass times, e.g. for tandem mill configurations. Metadynamic recrystallization has been effectively described using an Avrami equation, even though metadynamic recrystallization does not involve nucleation.⁷–⁹)

The difference between static recrystallization and dynamic or metadynamic recrystallization can be seen from the dependence of the recrystallization rate on the deformation parameters. As mentioned above, static recrystallization kinetics are strongly dependent on strain and temperature, and somewhat less so on strain rate. The dynamic or metadynamic recrystallization kinetics, however, are highly sensitive to strain rate and temperature, and not dependant on strain at all.

Recrystallization kinetics also depends on the steel composition. In this work, the steels have been microalloyed with V, and some have high Si levels. Published solubility data¹⁰) indicate that V is the most soluble of the conventional microalloying elements, and quantities in excess of 0.15% can be dissolved at normal reheating temperatures (e.g. 1200°C) regardless of carbon and nitrogen content.

In fact vanadium has a rather high solubility in austenite even at temperatures as low as 950°C. This is important for maximizing the precipitation strengthening effect, as the microalloying element must be completely dissolved at the reheating temperature and remain in solution until it precipitates in ferrite in the form of finely dispersed particles. As a consequence, vanadium in solid solution has little effect on the kinetics of recrystallization of the austenite of low carbon steels. Serajzadeh¹¹) studied the effect of silicon on austenite recrystallization behavior of C–Mn steels with silicon contents from 0.07 to 1.1 mass% at temperature 900–1200°C and strain rate of 0.01–1 s⁻¹. It was found that increasing the silicon content causes a retardation of kinetics of recrystallization of the austenite of low carbon steels.

The purpose of the present investigation is:

1. To examine the effect of vanadium and silicon additions on the kinetics of metadynamic recrystallization in high carbon steels by using compression tests.

2. To determine the conditions where metadynamic recrystallization is likely to predominate and to analyze the softening kinetics when the process involves both metadynamic and classical static recrystallization.
2. Experimental Procedure

2.1. Materials

The steels used in this work were prepared and hot rolled at the Materials Technology Laboratory, CANMET, (Ottawa, Ontario, Canada). The chemical compositions of the steels used in this study are given in Table 1. In this work, V was chosen as the key microalloying element rather than Nb, since it is more likely to precipitate in ferrite, leading to more effective precipitation strengthening. In addition, Si additions are of interest since Si has been linked with decreasing pearlite interlamellar spacing, leading to possible improvements in drawability.

To determine the austenitizing temperature at which V is fully dissolved prior to deformation testing, the following equations were used:

\[ \log[V] = \frac{9500}{T} + 6.72 \]  \hspace{1cm} (1)

\[ \log[V] = \frac{8330}{T} + 3.40 \]  \hspace{1cm} (2)

Table 2 gives the solubility temperatures of VN and VC calculated for the three steels used in this study. As noted previously, it shows that the solubility of both the nitride and the carbide are well below industrial steel reheating temperatures.

2.2. Experimental Equipment

Compression testing was carried out using an MTS (Materials Testing System) machine at the CSIRA (Canadian Steel Industry Research Association) Laboratory at McGill University, (Montreal, Quebec, Canada). Compression test specimens of 11.4 mm in height and an aspect ratio of 1.5 were machined from hot rolled plates with the longitudinal direction in the rolling direction. Details of the equipment are described elsewhere.

2.3. Thermal Cycles and Deformation Schedules

The following hot compression tests were performed in a radiant furnace using mica sheets lubricated with boron nitride, which were positioned between the anvils and the specimen, in order to reduce frictional effects. Prior to testing, all specimens were austenitized at 1 200°C for 20 min, and then slowly cooled (1°C/s) to the desired test temperature. After holding at the test temperature for 5 min, to equilibrate the specimen temperature, the deformation schedule was applied. Using this reheating procedure, the reheated austenite grain size was constant for all tests as shown in Table 1.

Table 2 gives the solubility temperatures of VN and VC calculated for the three steels used in this study. As noted previously, it shows that the solubility of both the nitride and the carbide are well below industrial steel reheating temperatures.

2.3.1. Single Hit Compression

To determine the dynamic recrystallization characteristics, the specimens were cooled to the test temperature (900, 950, 1 000, 1 050°C) held for 5 min to homogenize the temperature within the specimen. The specimens were deformed isothermally, to strains of up to 0.7, at a strain rate of 0.01, 0.1 or 1 s⁻¹, and the specimens were then water quenched.

2.3.2. Double Hit Compression Tests

For the metadynamic recrystallization tests, an initial deformation was performed to the peak strain of dynamic recrystallization, as determined by the previous single hit tests. Testing was conducted using a strain rate range of between 0.01 to 1 s⁻¹ and a temperature range from 900 to 1 050°C to generate a number of different dynamically recrystallized conditions. At the peak strain of dynamic recrystallization, the specimen was unloaded and held at the test temperature for times between 0.1 to 50 s to allow metadynamic recrystallization to occur. Then the specimen was reloaded to measure the level of metadynamic softening that had occurred and the specimen were water quenched.

The fractional softening, \( X \), is determined by:

\[ X = \frac{\sigma_2 - \sigma_1}{\sigma_1} \times 100\% \]  \hspace{1cm} (3)

where: \( \sigma_1 \) is the flow stress at the end the first hit, \( \sigma_2 \) is the offset stress (0.2%) of the first hit, and \( \sigma_1 \) is the offset stress (0.2%) of the second hit.

3. Results

3.1. Flow Curves

3.1.1. Single Hit Flow Curves

The flow curves obtained for the three steels deformed to a strain of 0.7 were plotted for the deformation temperatures (900–1 050°C) for various strain rates. All flow curves displayed a peak stress, although these were not well delineated at higher strain rates and lower temperatures as can be seen in Fig. 1 for steel A, for example.
strain curves with increasing unloading times are given in Fig. 2 for steel A, similar results being obtained with the other steels. As expected, when the unloading time is short, little softening occurs and, as a consequence, the flow curve of the second hit displays little work hardening prior to reaching the dynamic recrystallization peak. When the unloading time is increased, significant metadynamic recrystallization occurs, leading to increased work hardening on reloading. Note that the peak stress and strain are much smaller after full metadynamic recrystallization because of
the grain refinement that has occurred due to dynamic recrystallization. The higher grain boundary area per unit volume increases the recrystallization nucleation rate, since grain boundaries are preferred nucleation sites. This will therefore decrease the peak strain and consequently, the peak stress since less work hardening will have occurred in reaching the peak strain.

3.2. Effect of Deformation Parameters on the Softening

3.2.1. Effect of Strain Rate

In general, increasing the strain rate increases the flow stress, since less time is available for dynamic recovery. As well, there is a decrease in the subgrain size, and these two effects lead to an increase in the stored energy. Therefore, with increasing strain rate, the driving force for recrystallization will increase, and the recrystallization kinetics will be faster.

The effect of strain rate was investigated over the range 0.01 to 1 s\(^{-1}\) at 1050°C. The measured fractional softening is plotted as a function of the logarithm of the holding time as shown in Fig. 3 for steels A, B and C. For all the steels, these plots have the beginnings of a sigmoidal appearance, and, as anticipated, the recrystallization rate increases with increasing strain rate. For example, in steel A the time for 50% softening decreases from about 5 to less than 1 s as the strain rate is increased from 0.01 to 1 s\(^{-1}\). As compared with static recrystallization for the same steel A\(^{1o}\) the time for 50% softening decreases from about 80 to 30 s for the same strain rate change. The present observations are in good agreement with those reported by Hodgson\(^{16}\) and Roucoules.\(^{17}\)

3.2.2. Effect of the Deformation Temperature

The effect of deformation temperature on softening of metadynamic recrystallization is shown in Fig. 4 for steel B, and is typical of all the steels tested. In this case, the strain rate was held constant while the deformation temperature was varied. These results show that the rate of softening increases with increasing temperature.

3.2.3. Effect of Strain

The softening behavior shown in Fig. 5, which is typical of all the steels tested, indicates that strain has little influence upon the fractional softening of metadynamic recrystallization. This result agrees with those obtained by Hodgson,\(^{16}\) Roucoules\(^{18}\) and Bai.\(^{19}\)

4. Discussion

4.1. Modeling the Kinetics of Metadynamic Recrystallization

The kinetics of metadynamic recrystallization are usually described by an Avrami Eq. (5), which incorporates an em-
4.1.1. Determination of “n”

From Eq. (4), in order to determine $n$, the softening results were plotted as $\ln(\ln(1/X))$ vs. ln(time) plots. The results are presented for different conditions of temperature and strain rate, as shown in Fig. 6 for the three steels studied. In all cases, deformation was interrupted at the peak of dynamic recrystallization. The exponent $n$ was determined for the metadynamic recrystallization. The average values of $n$ were found to be 1.2, which approach the range of values of 1–1.6 observed by other workers.24–26) Values of $n$ for static and metadynamic recrystallization are gathered in Table 3. The current results are in good agreement with the average value of 1 found by Roucoules17) for Mo, Nb and Ti steels. The present value is also similar to the ones reported by Sellars23) for metadynamic recrystallization.

4.2. Determination of the Dependence of $t_{0.5}$ on the Deformation Parameters

4.2.1. Effect of Strain Rate

The strain rate exponent can be obtained from the $\ln(t_{0.5})$ vs. ln(strain rate) plots (Fig. 7). The average value of $p$ is $-0.6$, which is close to that obtained by Roucoules et al.18) for metadynamic recrystallization, and is somewhat lower than that observed by Hodgson et al.27) Elwazri et al.15,28) studied the effect of the strain rate on static and metadynamic recrystallization for high carbon and microalloyed steels. They found that metadynamic recrystallization exhibits a strain rate dependence about twice as strong as conventional static recrystallization as shown in Table 4. The present observation is in good agreement with previous work.29–31)

4.2.2. Activation Energy of Metadynamic Recrystallization

The activation energies were determined using the following Arrhenius relationship:

$$\ln\left(\frac{t_{0.5}}{Z^{0.6}}\right) = \ln(A) + \left(\frac{Q_{\text{MDRX}}}{R} \cdot \frac{1}{T}\right)$$

$$Z = \dot{\varepsilon} \exp\left(\frac{Q_{\text{act}}}{RT}\right)$$

where $\dot{\varepsilon}$ is the strain rate, $T$ is the absolute temperature, $R$ is the gas constant. $Q_{\text{act}}$ is the activation energy derived from the

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Table 3. Comparison of $n$ exponent for SRX and MDRX.

|         | Sellars [23] | Hodgson [24, 25] | Roucoules [17] | Elwazri [28] | Elwazri [19] | Present work |
|---------|--------------|------------------|----------------|--------------|--------------|--------------|
| MDRX    | 1            | 1.5              | 1              | 1.2          | -            | 1.2          |
| SRX     | 1-2          | 1                | -              | 1.3          | 1            | -            |

Table 4. Comparison of $p$ (strain rate) exponent for SRX and MDRX.

|         | Elwazri [28] | Elwazri [19] | Present work |
|---------|--------------|--------------|--------------|
| MDRX    | 0.6          | -            | 0.6          |
| SRX     | 0.34         | 0.4          | -            |
the steady state stress, and was found to be 350 kJ/mol for these microalloyed high carbon steels.\(^{14}\)

The parameter \(\ln\left(\frac{t_{0.5}}{Z^{0.6}}\right)\) is plotted as function of the inverse absolute temperature in Fig. 8. The activation energy for metadynamic recrystallization can be determined from the slopes and intercepts of these plots. A value of \(Q_{\text{MDRX}}=380\) kJ/mol was found for all three steels.

The activation energy in Eq. (8) is an apparent energy, which is a function of the activation energy of metadynamic recrystallization and, through the Zener–Hollomon parameter, also a function of the activation energy of deformation.\(^{6}\)

\[
Q_{\text{app}}=Q_{\text{MDRX}}-0.6Q_{\text{def}} \quad (8)
\]

\(Q_{\text{app}}\) is 170 kJ/mol, which is considerably lower than that for conventional static recrystallization as can be seen in Table 5. The present observation is in good agreement with previous work.\(^{29-32}\)

The effect of Si and V can be included in the Avrami type model through \(A_{\text{MDRX}}\), as shown in Fig. 9, and following linear relation was observed to hold

\[
A_{\text{MDRX}}=(1.5([\text{Si}]+[\text{V}])+2.8)\times10^{-8} \quad (9)
\]

Thus, the dependence of \(t_{0.5}\) on Si+V concentrations, Zener–Hollomon parameter, strain rate and deformation temperature can be expressed as follows:

\[
f_{0.5} = \frac{170}{RT} \exp\left(\frac{170}{RT}\right)
\]

\[
A_{\text{app}} = A_{\text{MDRX}} \quad (10)
\]

A similar type of expression has been generated for the classical static recrystallization of these steels.\(^{15}\)

A comparison between the predicted (with the effect of \([\text{Si}+\text{V}]\) on \(A_{\text{MDRX}}\)) and measured values of \(t_{0.5}\) for both classical static and metadynamic cases\(^{15,28}\) is made in Fig. 10. This can be compared with Fig. 11, in which \(A_{\text{MDRX}}\) has not been adjusted for V and Si for both static and dynamic recrystallization. As can be seen, the effect of Si and V is significant.

To the knowledge of the author, this is the first attempt to quantify the effect of V and Si on static and metadynamic recrystallization.

It is generally accepted that the effect of the alloying and microalloying elements on retarding the onset of metadynamic recrystallization is related to the atomic size difference between \(\gamma\)-Fe and alloying elements; e.g. Mn, Si, V, Mo, Ti and Nb, which increases in the order listed. The effect of Mn could not be determined for these alloys, but it generally plays only a minor role in retarding recrystallization.\(^{40}\)
tion due to its similar atomic size and diffusion rate compared to iron.

4.3. Effect of Strain on Softening Behavior

The effect of strain on fractional softening at different strain rates is shown in Fig. 12. As expected, fractional softening increases up to a certain strain, \( \varepsilon_T \), the transition strain. The fractional softening plateau that occurs beyond \( \varepsilon_T \) is due to full metadynamic recrystallization, which is independent of the applied strain. The key data of these graphs regarding the static softening, the critical strain as well as the transition strain are summarized in the Table 6.

The values for strain transition are approximately a factor of 1.5 higher than the peak strain. This is the same as that obtained by Bai et al. for two different Nb microalloyed steels.33)

4.4. Modeling the Static Softening

The effect of strain on static recrystallization can be modeled by considering the existence of the following three recrystallization regimes, (i) pure classical static, (ii) mixed classical and metadynamic and (iii) pure metadynamic recrystallization, as indicated by Luton et al.34) Bai et al.33) and Uranga et al.3) These three regions are schematically indicated in Fig. 13, which indicates the recrystallization mechanisms that correspond with the illustrated levels of applied strain.

To separate the softening behavior observed into these three regions, empirical equations can be fitted to the individual softening processes, and these can be used to model the static kinetics in each region. The empirical equations used to model the softening in the three different regions are indicated in the following:

4.4.1. Region I: \( \varepsilon < \varepsilon_T \)

In this region, recrystallization is due to classical static recrystallization. The following equations for specifying \( t_{0.5} \) and \( X \) in vanadium steels have been formulated to model this region for these steels14):

\[
X_{SRX} = 1 - \exp \left( -0.693 \frac{f}{t_{0.5,SRX}} \right) \quad \text{...(11)}
\]

\[
t_{0.5,SRX} = \frac{0.4([V]+[Si])+1.1 \times 10^{-17} \varepsilon^{-2}d_0^{2}e^{-0.4} \exp \frac{290 \times 10^{3}}{RT}}{\text{...(12)}}
\]

where \( d_0 \) is the initial austenite grain size and \([V]\) and \([Si]\) represent the amount of vanadium and silicon in solution in mass%.

4.4.2. Region III: \( \varepsilon > \varepsilon_T \)

This region is due to metadynamic recrystallization during the interpass intervals. The equations for specifying \( t_{0.5} \) and \( X \) in vanadium steels are Eqs. (4) and (10).

4.4.3. Region II: \( \varepsilon_T < \varepsilon < \varepsilon_T \)

In this region both static and metadynamic recrystallization take place. Thus, the total fractional softening due to both processes can be given by the sum of the individual components34)
When the static softening is completed

\[ X'_F = X^{SRX}_F + X^{MDRX}_F = 1 \]  

(14)

where the subscript "F" refers to the final contribution of the corresponding mechanism to the completed recrystallization. The kinetics of recrystallization involving both static and metadynamic mechanisms can be evaluated using the corresponding Avrami equations:

\[ X^{SRX}_F = X^{SRX}_F \left[ 1 - \exp \left( -0.693 \left( \frac{t}{t^{SRX}} \right)^{n} \right) \right] \]  

(15)

\[ X^{MDRX}_F = X^{MDRX}_F \left[ 1 - \exp \left( -0.693 \left( \frac{t}{t^{MDRX}} \right)^{n} \right) \right] \]  

(16)

(The strain used to calculate the amount of classical static recrystallization when the applied strain is between the critical and transition strains is taken to be the critical strain.)

Thus, for \( t \) approaching \( \infty \), the mechanisms approach their corresponding maximum values. A simple approximation for the metadynamic mechanism is

\[ X^{MDRX} = \frac{\varepsilon - \varepsilon_c}{\varepsilon_a - \varepsilon_c} \]  

(17)

when \( \varepsilon \leq \varepsilon_c \), the contribution of metadynamic recrystallization is zero.

4.4.4. Comparison of the Empirical Restoration Kinetics with Experimental Data

The results are presented as plots of fractional softening vs. strain for a fixed time periods, as shown in Fig. 14. The solid line is the prediction based on the experimental constants determined above. The individual data points are the softening measurements obtained from the mechanical technique. There is a very good agreement between the prediction and the experimental data of softening generated for these steels from all of the tests described above.

The model was also used to describe the time for 50% softening, \( t_{0.5} \), for the strain rates of 0.01 and 1 s\(^{-1}\) with \( D_0 = 94 \mu m \). When the strain rate increased from 0.01 to 1 s\(^{-1}\), \( t_{0.5} \) decreased from 15 to 0.8 s and 3 to 0.3 s for static and metadynamic recrystallization, respectively. The \( t_{0.5} \) values for critical strains for the strain rates of 0.01 s\(^{-1}\) and 1 s\(^{-1}\) are 0.13 and 0.3. There is a transition region between the critical strain and transition strain where the both static and metadynamic recrystallization softening co-exist during the inter-pass time. Figure 15 clearly shows three distinct regions (the static and metadynamic recrystallization kinetics), experimentally, which is well predicted by this analysis.

5. Conclusions

Three steels containing different amounts V and Si were tested using the double hit compression testing. A kinetic model has been proposed which takes the V and Si concentrations into account. A model incorporating three-static
recrystallization regimes has been used to describe the restoration curves obtained during the hot working of steels. According to this model, when strained into the partial dynamic recrystallization region, the subsequent static softening arises from a linear combination of classical static and metadynamic recrystallization.

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