Static recrystallization kinetics of ferrite in cold-deformed medium carbon steel

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Abstract
Static recrystallization (SRX) kinetics of ferrite in cold-deformed medium carbon steel (AISI 4130) at subcritical temperatures was studied for proposing a comprehensive Johnson–Mehl–Avrami–Kolmogorov (JMAK) equation. The value of the recrystallization activation energy ($Q_{SRX}$) was near the activation energy for lattice diffusion in α-iron (251 kJ mol$^{-1}$). Therefore, the atomic mechanism for SRX of ferrite in this material was considered to be the self-diffusion in α-iron. It was revealed for the first time that the pre-exponential constant ($K_0$) in the equation of the overall rate constant ($K$) depend exponentially on the reduction in thickness ($r$). The Avrami exponent ($n$) of $\sim 1$ was determined, which was interpreted in terms of the nucleation of recrystallization on grain boundaries. Accordingly, a JMAK equation with incorporation of the effects of annealing temperature and reduction in thickness was proposed, which can be used for grain refinement of this material for industrial applications.

1. Introduction

Some automobile parts for steering, engines, suspensions, and transmissions are made from medium carbon steels [1]. With carbon content in the range of 0.25 to 0.55 wt%, the quench/temper heat treatment is usually used for strengthening of these steels [1, 2].

Another common route for strengthening of engineering materials is grain refinement, which can be realized via recrystallization processes [3–6]. The behaviors of medium carbon steels during dynamic (DRX), metadynamic (MDRX), and static (SRX) recrystallization have been investigated. Ebrahimi et al [7], Cabrera et al [8], Zhang et al [9], Gu et al [10], Chen et al [11], Wang et al [12], and Saadatkia et al [13] studied the hot forming and DRX behaviors of steels with 0.27, 0.34, 0.36, 0.43, 0.45, and 0.50 wt% C, respectively, where the great potentials of hot working for processing of these steels were revealed. Bianchi and Karjalainen [14] and Lin et al [15] studied the MDRX behavior of medium-carbon steels following hot deformation. Moreover, the SRX behaviors of medium-carbon steels in the austenitic regime have been studied by Carsi et al [16], Ayada et al [17], Medina et al [18], Lin et al [19], Opiela and Grajcar [20], and Opiela and Ozgowicz [21]. Alaneme [22] studied the intercritical annealing of a cold deformed medium carbon steel, where recovery processes, primary recrystallization, intense spheroidization, ferrite to austenite transformation, and grain growth were identified by hardness test.

Besides those excellent works, the reported works on the SRX behavior in the ferritic regime are scant [23, 24]. Herrera et al [23] studied the cold rolling behavior of 1050 steel and they also investigated the texture evolution as a result of annealing at 700 °C for 13 h. The SRX behavior during continuous heating for the cold rolled AISI 4130 medium carbon steel and the response of this steel to intercritical annealing treatment were studied by Tavakoli et al [24], which was characterized by an initial drop of hardness due to recrystallization and subsequent rise of hardness as a result of austenitization.
However, an important subject from the industrial and scientific standpoints, i.e. SRX kinetics, for the medium carbon steels has been remained to be unraveled. As a result, the present work is dedicated to study the SRX kinetics of a commercial medium-carbon steel (AISI 4130) at subcritical temperatures for proposing an Avrami equation by consideration of reduction in thickness ($r$) in the proposed formula.

2. Experimental material and procedures

AISI 4130 steel with chemical composition (wt%) of 0.27C-0.71Mn-0.25Si-0.94Cr-0.15Mo was received in the annealed ferrite/pearlite condition. A cylindrical specimen with diameter of 2 mm and length of 10 mm was heated up to 900 °C at the rate of 2 °C s$^{-1}$ to obtain the dilatometric curve of figure 1(a). With the aid of the derivative curve, the lower critical temperature ($A_1$) was determined as 727 °C. The as-received sheet was then cold rolled with reduction in thickness ($r$) of 0.35, 0.50, 0.70, and 0.90. Then the cold rolled sheets were subcritically annealed for 1 h at temperatures in the range of 550 °C and 720 °C. The choice of 720 °C as the upper bound for thermal treatments was based on the $A_1$ temperature of 727 °C. Moreover, the 35% and 90% rolled sheets were also annealed at 670 °C for holding times in the range of 15 min and 3 h. The choice of 670 °C as the annealing temperature will be discussed later. The applied routes are schematically shown in figure 1(b).

Etching in the 2% Nital solution and an optical microscope were used for revealing the microstructures. Vickers hardness test with a load of 5 kg was used to study the softening during SRX. The error bars were not shown on the hardness curves for the sake of brevity. However, it is declared that the standard deviations of the hardness measurements were less than 4 HV.

3. Results and discussion

3.1. Annealing for 1 h at different temperatures

Figure 2(a) shows the evolution of hardness of the cold rolled sheet during annealing for 1 h at different temperatures. It can be seen that the hardness of the cold rolled sheets (at room temperature, RT) increases with increasing $r$. This can be related to the effect of cold rolling on the as-received sheet (figure 3(a)), where the grains became more pancaked at $r$ of 0.9 (figure 3(c)) compared to $r$ of 0.35 (figure 3(b)).

At each $r$, it can be seen in figure 2(a) that up to 600 °C, the hardness does not change considerably. Afterwards, the hardness drops rapidly and reaches a minimum value at 720 °C. This fall is related to the progression of SRX as can be seen in figures 3(d) and (e), where incomplete recrystallization can be seen at 670 °C (figure 3(d)) while fine and recrystallized one can be seen at 720 °C (figure 3(e)).

On the other hand, the minimum hardness at 720 °C (figure 2(a)) increases with increasing $r$. This is related to the effect of $r$ on the grain size of recrystallized microstructures. This can be verified by comparing the recrystallized microstructure related to $r$ of 0.35 (figure 3(e)) to that of 0.9 (figure 3(f)), where the average ferrite grain size of the former and the latter was determined as 8.6 μm and 4.9 μm, respectively. Both of these grain sizes are much finer than the average grain size of the as-received sheet, which was determined to be 28 μm (figure 3(a)).
The recrystallized fraction \( X \) can be calculated based on equation (1) \([25]\):

\[
X = 1 - \frac{(H - H_m)}{(H_M - H_m)}
\]  

where the maximum hardness \( H_M \) and the minimum hardness \( H_m \) are related to the cold rolled state and 720 °C, respectively. The plots of \( X \) versus \( T \) are depicted in figure 2(b), where sigmoidal (S-shaped) curves can be seen. Therefore, the Johnson–Mehl–Avrami–Kolmogorov (JMAK) \([25–27]\) analysis should be applied to study the SRX kinetics. Meanwhile, it can be seen that the SRX kinetics are faster for higher \( r \), which can be realized by observing the shift to the left side with increasing \( r \). Moreover, the time corresponding to the recrystallized fraction of \( X = 0.5 \) is smaller for higher \( r \) values. This is related to the higher stored energy and higher driving force for recrystallization with increasing the amount of deformation \([28]\).

### 3.2. Determination of recrystallization activation energy \( Q \)

In the JMAK relation of equation (2), the Avrami exponent \( n \) and the overall rate constant \( K \) are related to the nucleation mode and the transformation rate, respectively:

\[
X = 1 - \exp(-Kr^n)
\]  

For the holding time of 1 h, the term \( r^n \) is removed. Therefore, equation (3) and then equation (4) can be obtained:

\[
X = 1 - \exp(-K)
\]  

\[
K = \ln\{1/(1 - X)\}
\]  

The parameter \( K \) can be expressed as follows \([29]\):

\[
K = K_0 \exp(-Q_{SRX}/RT)
\]  

where \( K_0 \) is a constant and \( Q_{SRX} \) is the recrystallization activation energy. Now, based on equations (4) and (5), the following relation can be obtained by taking natural logarithm:
Therefore, as shown in figure 4(a), the slope of the plot of \( \ln (1/(1 - X)) \) can be used for obtaining the value of \(-Q_{SRX}/R\).

It can be seen in figure 4(a) that straight lines can represent the data, and hence, the slope of these lines were taken for obtaining the values of \(Q_{SRX}\) for different values of \(r\). The obtained values are shown in figure 4(a), where it can be seen that these values are relatively consistent. More importantly, these values are near the activation energy for lattice diffusion in \(\alpha\)-iron (251 kJ mol\(^{-1}\) [30, 31]). Therefore, the micro-mechanism of SRX in this material is the self-diffusion in \(\alpha\)-iron.

Accordingly, the \(Q_{SRX}\) value of 251 kJ mol\(^{-1}\) (and hence the slope of \(251000/R = 30190\)) was used for obtaining the values of \(\ln K_0\) from the slopes of the curves in figure 4(b). Subsequently, the obtained values were plotted against \(r\) in figure 4(c). It can be seen that, by increasing \(r\), the value of \(\ln K_0\) increases. A relation of the form of equation (7) was achieved, which quantitatively demonstrates the effect of cold rolling reduction (\(r\)) on the recrystallization kinetics:

\[
\ln (1/(1 - X)) = \ln K_0 - (Q/R)(1/T)
\]

3.3. Annealing at 670 °C for different holding times

As mentioned in the experimental section, the cold rolled sheets were also annealed at 670 °C for holding times in the range of 15 min and 3 h. The choice of 670 °C as the annealing temperature can be realized by consideration of figure 2(b). It can be seen that at \(\sim 670^\circ\)C all of the curves reach the recrystallization faction of \(~0.5\) at holding time of 1 h. Therefore, the recrystallization temperature of this material can be considered to be \(\sim 670 \^\circ\)C despite the fact that it has a dependency of \(r\) (with increasing \(r\), the recrystallization temperature declines). Therefore, this temperature was considered for kinetics analysis for the \(r\) values of 0.35 and 0.90.

Figure 5(a) shows the evolution of hardness of the cold rolled sheets during annealing at 670 °C. At \(r = 0.90\), it can be seen that the hardness declines during annealing and reaches a minimum value at 2 h. It can be seen in the figure that this minimum value is consistent with the minimum value obtained from figure 2(a) for \(r = 0.90\). The same trend of hardness fall can be also seen for \(r = 0.35\), except the fact that at 3 h, the plateau of hardness

![Representative microstructures: (a) as-received sheet, (b) 35% rolled sheet, (c) 90% rolled sheet, (d) 35% rolled sheet annealed at 670 °C for 1 h, (e) 35% rolled sheet annealed at 720 °C for 1 h, and (f) 90% rolled sheet annealed at 720 °C for 1 h.](image-url)

\[K_0 = \exp(0.76r + 31.3)\]
has not reached. This reveals the slower SRX kinetics for \( r = 0.35 \) compared to \( r = 0.90 \). Therefore, for calculation of \( X \), the minimum value obtained from figure 2(a) for \( r = 0.35 \) was considered. The line corresponding to the latter is also shown in figure 5(a). It can be seen that more holding time is required to reach the complete recrystallization corresponding to the hardness plateau.

3.4. Determination of Avrami exponent (n)
The JMAK relation of equation (2) results in the following relation:

\[
\ln \{ \ln \left[ 1 / (1 - X) \right] \} = \ln K + n \ln t
\]

Therefore, the slope and the intercept of the plot of \( \ln \{ \ln \left[ 1 / (1 - X) \right] \} - \ln t \) can be used for obtaining the values of \( n \) and \( \ln K \), respectively. These plots are shown in figures 5(b) and (c). It can be seen that the data can be represented by a line, where the slope of this line is \( n = 0.977 \) for \( r = 0.35 \) and \( n = 1.23 \) for \( r = 0.90 \). The predefined values of \( n \) in the JMAK kinetic law are 1, 2, 3, 3-4, 4, and >4 for grain boundary nucleation after saturation, grain edge nucleation after saturation, zero nucleation rate (satisfaction of point sites), decreasing nucleation rate, constant nucleation rate, and increasing nucleation rate, respectively [32]. Therefore, based on the obtained values of 0.977 and 1.23, it can be deduced that the Avrami exponent (\( n \)) for SRX of ferrite is \( \sim 1 \), which can be interpreted in terms of the nucleation of recrystallization on grain boundaries [32–34]. Nucleation of recrystallization on grain boundaries was an expected result [28], which was quantitatively shown in the present work.

For checking the kinetics analysis, the \( \ln K_0 \) values obtained from figure 5 (calculation of \( \ln K_0 \) from \( \ln K \)) by consideration of Avrami exponent of 1 can be compared to those determined from figure 4(b) by consideration of SRX activation energy of 251 kJ mol\(^{-1}\). Based on figure 5, the \( \ln K_0 \) values of 30.91 and 31.62 were determined for \( r \) of 0.35 and 0.90, respectively. These are relatively consistent with the determined values from figure 4(b) (31.55 and 31.90 for \( r \) of 0.35 and 0.90, respectively). Therefore, these two approaches are consistent.
Based on the analysis of figures 4 and 5, the following relation can be proposed for SRX of ferrite in medium carbon steel:

\[ X = 1 - \exp(-Kr^t) \]
\[ K = K_0 \exp(-251000/RT) \]
\[ K_0 = \exp(0.76r + 31.3) \]  \hspace{1cm} (9)

4. Summary

In summary, the static recrystallization (SRX) kinetics of a commercial medium carbon steel (AISI 4130) at subcritical temperatures was studied for proposing a Johnson–Mehl–Avrami–Kolmogorov (JMAK) equation. The values of recrystallization activation energy were near the activation energy \( (Q_{SRX}) \) for lattice diffusion in \( \alpha \)-iron \((251 \text{ kJ mol}^{-1})\). Therefore, the micro-mechanism for SRX of ferrite in this material was considered to be the self-diffusion in \( \alpha \)-iron. It was revealed for the first time that the pre-exponential constant \( (K_0) \) in the equation of the overall rate constant \( (K) \) depend exponentially on the reduction in thickness \( (r) \). The Avrami exponent \( (n) \) of \( \sim 1 \) was determined, indicating the nucleation of recrystallization on grain boundaries. Accordingly, the JMAK equation of \( X = 1 - \exp\{ -\exp(0.76r + 31.3)\exp(-251000/RT)\}t \) with incorporation of the effects of annealing temperature \( (T) \) and reduction in thickness \( (r) \) can be proposed for grain refinement of this material. Quantitative investigations of the effects of processing parameters on the obtained grain size and crystallographic texture are suggested for the future works that are based on the EBSD and TEM methods.

Figure 5. (a) Hardness evolution during annealing at 670 °C, (b) Plot used for obtaining \( n \) and \( \ln K \) for \( r = 0.90 \), and (c) Plot used for obtaining \( n \) and \( \ln K \) for \( r = 0.35 \).
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