Microstructural Evolution and Kinetics for Post-dynamic Transformation in a Plain Low Carbon Steel

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(Received on December 17, 2007; accepted on April 17, 2008)

The post-dynamic transformation (post-DT), which could occur during isothermal holding after hot deformation, was investigated by using both dilatometry method and optical microstructural observation in a plain low carbon steel. The results indicates that the kinetics of post-DT at deformation temperature between Ae₃ and Ar₃ can be well described by the Avrami equation: \(X = 1 - \exp(-kt^n)\), but the \(n\) value is lower than that of the corresponding static transformation due to the early impingement of formed ferrite grains. Furthermore, the ferrite-to-austenite re-transformation was measured by dilatometry during the isothermal holding after hot deformation above Ae₃ temperature, which suggests that dynamic transformation can indeed occur even above Ae₃.

KEY WORDS: post-dynamic transformation; kinetics; microstructure; low carbon steel; dilatometry.

1. Introduction

The key concept of the conventional thermomechanical controlling process (TMCP) commonly employed in steel industries is to apply a large deformation in the non-recrystallized region so that the maximum nucleation sites are achieved by introduction of pan-caked grain boundaries and deformation bands in the austenite, which can then transform to fine ferrite grains down to 5 \(\mu\)m during the cooling after the finish rolling.\(^{11}\) Recently, the dynamic austenite-to-ferrite transformation (DT) in low carbon steel, \(i.e.\) transformation occurs during deformation applied in the temperatures above Ar₃, has received much wider attention because the ferrite grain size can be refined to about 1 \(\mu\)m in microalloyed steel and to about 3 \(\mu\)m in plain low carbon steel.\(^2\)–\(^6\) Therefore, this is a very promising production process for ultrafine grained steels to achieve better strength and toughness simultaneously. In this new production route, some amount of ferrite has been formed during the deformation, and thus, the remaining deformed austenite will continue to transform to ferrite while the formed ferrite grains get coarsening during the inter-pass period or post-deformation cooling. Kinetics of such post-dynamic transformation after deformation could be different from that in the conventional TMCP because it could only involve a process of ferrite grains growing into the parent austenitic phase without the nucleation stage, which is similar to the phenomena of meta-dynamic recrystallization commonly observed in the high-speed hot rolling production line. Even if DT has been intensively studied as an emerging new technology to produce ultrafine grained steels, it is surprising to find that the post-dynamic transformation has received little attention, although such transformation definitely has a very important influence on the final microstructure in the product. Therefore, in this paper, we investigated the isothermal post-dynamic transformation after deformation applied both above Ae₃ and between Ae₃ and Ar₃.

2. Experimental Procedures

2.1. Specimens Preparation

The chemical compositions of the investigated low carbon steel are 0.17C–0.27Si–0.71Mn (wt%). The steel was melted in the form of 25 kg ingot in a vacuum induction furnace. The ingot was forged into a long rectangular bar after soaking treatment at 1 473 K for 1 h, and then a small portion of the rectangular bar was sectioned and then further forged into near-cylindrical bar with diameter of 15 mm. Next, the near-cylindrical bar was homogenized at 1 573 K for about 2 h to ensure the uniform composition. Cylindrical specimens of 100 mm in total length with threaded ends and with gauge length 10 mm and gauge diameter 9 mm in the middle, which were used for thermo-mechanical simulation, were machined from the near-cylindrical bar.

2.2. Determination of Transformation Temperature

The transformation temperature of Ae₃ of the investigated steel was calculated by Thermo-Calc software with database “TCFE3” as 1 108 K, and Ar₃ is 983 K, measured by dilatometry in the cooling rate of 20 K/s from solution at 1 273 K for 0.3 ks. In order to verify the calculated Ae₃ temperature, the samples, which were same to the ones used for
thermo-mechanical simulation, were soaked in an electrical resistance at various temperatures around $A_e$ for 2 h, and then quenched into water. The range of temperature fluctuation in the furnace was ±1 K during the soaking. The microstructures of the water-quenched samples are given in Fig. 1. It can be seen that the microstructure is fully composed of martensite when the soaking temperatures is 1113 K or higher, indicating that the steel was fully austenitized at or above 1113 K. At 1103 K, a small amount of ferrite can be observed, clearly indicating that the steel was not fully austenitized at this temperature. In addition, the volume fraction of ferrite increases when the soaking temperature decreases from 1103 to 1093 K. Thus, it can be concluded that the $A_e$ temperature of the tested steel is between 1113 K and 1103 K. Therefore, the calculated $A_e$ temperature (1108 K) by Thermo-Calc is considered to be reliable.

2.3. Thermo-mechanical Simulation Tests

The thermo-mechanical simulation tests were performed on a Gleeble3800 thermo-mechanical simulator. The thermo-mechanical simulation schedule was employed as shown in Fig. 2. The specimen was heated up to 1273 K with a heating rate of 10 K/s and held for 180 s to obtain single austenitic microstructure. The specimen was then cooled to various temperatures with a rate of 20 K/s, held for 5 s to stabilize the temperature and then deformed in compression to various true strains at a strain rate of 1 s$^{-1}$. After deformation, the specimens were unloaded completely within 0.2 s. Then, the specimens were isothermally held for 1.2 ks followed by gas cooling. The curves of true stress vs. true strain during deformation were derived by a direct measurement of diametrical change of the specimen during deformation via an attached LVDT quartz dilatometer. The change of diameter during the isothermal holding was also monitored and recorded. During the testing the specimens were protected from oxidation by a vacuum at the order of $10^{-5}$ MPa. It should be mentioned that the high-resolution quartz dilatometer must be carefully attached to the specimen so that the welding points of the thermocouple wires for temperature control and the contact points between the dilatometer and the specimen are all at the same cross section, which ensures that the measured diameter change indeed occurs at the measured temperature. The diametric temperature gradient in the specimen with 9 mm diameter is generally quite small in the high vacuum and thus it can be ignored. In order to investigate the microstructural change during isothermal holding, some specimens were water quenched immediately after deformation or after some isothermal holding. The microstructures of the water-quenched specimens were observed under optical microscope after proper etching.

3. Results and Discussion

3.1. Determination of Critical Strain for DT

The prerequisite for studying post-DT is to know the critical strain for DT. There is a simple method to determine
the critical strain for the occurrence of DT or dynamic recrystallization (DRX) by using stress–strain curves.\(^{24,38}\)

The initiation of DRX or DT corresponds to the following condition:

\[
\frac{\partial}{\partial \sigma} \left( \frac{\partial \theta}{\partial \sigma} \right) = 0 \quad \text{(1)}
\]

where \(\theta\) is the conventional strain hardening rate, \(\frac{\partial \sigma}{\partial \varepsilon}\).

Equation (1) means that the initiation of DRX or DT corresponds to the inflection point in the \(\theta–\sigma\) curve and the minimum point in the \(-\left(\frac{\partial \theta}{\partial \sigma}\right)–\sigma\) curve. Figure 3(a) shows the true stress vs. true strain curves of the tested steel at various deformation temperatures and at the strain rate of 1 s\(^{-1}\). The \(\theta–\sigma\) curves and \(-\left(\frac{\partial \theta}{\partial \sigma}\right)–\sigma\) curves can be obtained from the stress–strain curves and are shown in Fig. 3(b) and Fig. 3(c) respectively. From Fig. 3(c), the critical strain for DRX or DT can be determined, as shown in Fig. 3(d). It can be seen that in the temperature region below 1 133 K, the critical strain decreases as deformation temperature is lowered, but a reversed tendency is exhibited in the temperature region above 1 133 K. It is well documented that the critical strain for DRX increases with increasing Zener–Hollomon parameter whereas the critical strain for DT decreases with decreasing temperature due to larger driving force and higher nucleation rate at lower temperature.\(^{24}\) Therefore, dynamic transformation occurs in the temperature region below 1 133 K and dynamic recrystallization occurs at temperatures higher than 1 133 K. It is noted that the derivative analysis (in Fig. 3) indicates that dynamic transformation can even occur at above \(A_e\) temperatures (1 108 K). This will be further confirmed by the dilatometry measurement and microstructural observation presented in the following text.

3.2. Post-dynamic Transformation at Temperatures below \(A_e\)

Figure 4 shows dilation curves of the specimens during the isothermal holding after deformation to various strains at 1 023 K.

**Fig. 3.** Determination of the critical strain for DRX or DT. (a) True stress vs. strain curves at various deformation temperatures; (b) \(\theta–\sigma\) curves; (c) \(-\left(\frac{\partial \theta}{\partial \sigma}\right)–\sigma\) curves and (d) critical strain for DRX or DT as a function of deformation temperature.

**Fig. 4.** Dilation curves of the specimens during the isothermal holding after deformation to various strains at 1 023 K.
Figure 5 shows the microstructures of the specimens obtained by water quenching after different periods of isothermal holding at 1 023 K without deformation. Figure 6 illustrates the microstructures at the center of the specimens deformed to various strains and then subjected to different periods of holding. It can be seen from Fig. 5 that the ferrite grains are mainly nucleated at the triple points of the prior-austenite grains at the beginning, then grow up freely and finally impinge with each other at least in two-dimensions. Because the nucleation sites are limited in this case, the final ferrite grain size is very large. Just immediately after deformation, fine ferrite grains with the size of about 3 μm are found and more ferrite grains formed at larger strain, as shown in Fig. 6(a) and Fig. 6(d). Furthermore, these dynamically transformed ferrite grains are mainly nucleated at the prior-austenite grain boundaries at a relatively low strain (ε=0.40), but nucleated both at the austenite grain boundaries and in the interior of austenite grains at a relatively large strain (ε=0.64). After the isothermal holding, both the grain size and volume fraction of ferrite increase due to the post-dynamic austenite-to-ferrite transformation and grain coarsening. It is worth mentioning that the transformation fraction, X, is not the real ferrite volume fraction in this case. Since Avrami equation requires the transformation fraction, X, varying from 0 to 1, and we only focus on the post-dynamic transformation kinetics during the isothermal holding, the transformation fraction X is defined by

\[ X = \frac{f_{v_{\text{iso}}}^\text{end} - f_{v_{\text{iso}}}^\text{start}}{f_{v_{\text{iso}}}^\text{end} - f_{v_{\text{iso}}}^\text{start}} \] ..................................................(3)

where \( f_{v_{\text{iso}}}^\text{start} \), \( f_{v_{\text{iso}}}^\text{end} \) and \( f_{v_{\text{iso}}}^\text{iso} \) are the ferrite fractions at the beginning, moment t and end of the isothermal holding. \( f_{v_{\text{iso}}}^\text{end} \) is considered to be quite close to the equilibrium ferrite fraction at the test temperature. In our tests, X-time curves in Fig. 7(a) can be directly obtained from the dilatation–time curve during the isothermal holding (Fig. 4) because the dilation is proportional to the formed ferrite fraction in the sample.\(^{29}\)

The Avrami exponent is generally derived from the slope of \( \log[-\ln(1/(1-X))] \) versus \( \log(t) \) plot by the linear regression as shown in Fig. 7(b). It is evident that n value de-
creases and $k$ value increases as the applied strain increases. The Avrami exponent $n$ can be influenced by the mechanisms of nucleation and growth,\(^{39}\) so the value of $n$ can be used to analyze the physical meaning of nucleation and growth mechanism behind the transformation.\(^{39}\) For example, a diffusion controlled ferrite transformation with the “site-saturation” nucleation mechanism and parabolic growth rate should have the time exponent $(n)$ value of 1.5, 1 and 0.5 for three, two and one-dimensional growth, respectively. For static transformation without deformation, most of grains can grow without impingement in three-dimensions at the beginning of the transformation, so $n$ value is close to 1.5, and then it gradually decreases due to the impingement between more ferrite grains formed, which is exactly same to the observation shown in Fig. 7(b). When dynamic transformation has taken place during deformation, most of prior austenite grain boundaries are covered by dynamically transformed ferrite and therefore the ferrite transformation during the subsequent holding is just ferrite growth into the austenite grain interiors. As a consequence, the high value of $n$, which is observed at the beginning of static transformation, can not appear during the post-dynamic transformation. Instead, ferrite grains grow into the austenite grain interior with just one dimension at the beginning of post-DT due to the edge impingement between the dynamically formed ferrite grains. Therefore, the Avrami exponent $n$ of post-dynamic transformation is determined by both long-range diffusion-controlled growth and impingement of the formed ferrite grains. A larger strain can lead to more ferrite formed dynamically during deformation and then impingement can occur at the earlier stage of transformation, particularly when more intragranular ferrite is formed at the larger strain. This is the possible reason why the Avrami exponent of $n$ during post-dynamic transformation is smaller than that during the static transformation, and larger strain leads to smaller value of $n$. However, further effort is still required to quantify the complicated relationship between the applied strain and the exponent $n$.

3.3. Post-dynamic Transformation at Temperatures above $\text{Ae}_3$

Figure 8 shows the dilation curves of the specimens during isothermal holding after deformation at various temperatures above $\text{Ae}_3$. At 1 223 K, the diameter of the specimen remains almost constant during isothermal holding, indicating that no transformation occurs. At both 1 113 K and 1 113 K, however, the specimens contract clearly at the beginning 200 s of the isothermal holding, suggesting that a ferrite-to-austenite transformation may take place. The only reasonable explanation for this is that the dynamic austenite-to-ferrite transformation has taken place during deformation at above $\text{Ae}_3$ temperature, and the formed ferrite may then re-transform to the austenite during the subsequent isothermal holding because ferrite is not thermodynamically stable above $\text{Ae}_3$ temperatures when the stored energy in the deformed austenite is diminished due to recovery. It should be noted that during thermo-mechanical treatment using Gleeble machine, temperature might drop locally very shortly due to chilling effect by anvils. Figure 9 shows the temperature change during deformation at 1 113 K and 1 113 K. It can be seen that the temperature dropped during deformation indeed, and then rose to the preset temperature in 3 s during the subsequent holding. In the case of deformation at 1 113 K, the temperature dropped from 1 113 to 1 098 K, which is below the $\text{Ae}_3$ temperature (1 108 K). But in the case of deformation at 1 133 K, although the temperature dropped from 1 133 to 1 113 K, the temperature is still above $\text{Ae}_3$. In the later case, the deformation is always imposed in the temperature range above $\text{Ae}_3$, however, the shrinkage of the sample can still be detected, indicating that the dynamic transformation can occur even above $\text{Ae}_3$ temperature indeed. This was confirmed by the microstructural observations too, as shown in Fig. 10. A considerable amount of ferrite, mostly nucleated at austenite grain boundaries, can be observed on the specimen immediately quenched after compression to the true strain of 0.69 at 1 123 K with the strain rate of $1 \text{s}^{-1}$, but it could not be observed after 1.2 ks isothermal holding fol-

![Fig. 7. Isothermal post-dynamic transformation kinetics curves (a) and the plot of $\log\left[\frac{-\ln(1-X)}{t}\right]$ versus $\log(t)$ for various strains at 1 023 K.](image)

![Fig. 8. Dilation curves of the specimens during isothermal holding after deformation at various temperatures above $\text{Ae}_3$.](image)
lowing deformation.

To date, it is controversial whether DT can occur at temperatures above $A_{E3}$. For example, Liu et al.\textsuperscript{32,33} found, by means of optical microscope observation on the water-quenched samples, the formation of dynamically transformed ferrite in the 0.15C–0.19Mn–0.49Si (wt\%) steel deformed at 1 123 K (the $A_{E3}$ is 1 121 K) with a reduction of 80\% and the re-transformation from ferrite to austenite during subsequent holding. In contrast, Choi et al.\textsuperscript{24} ascertained also by means of optical microscope observation on the water-quenched samples that DT cannot occur above $A_{E3}$ temperatures in the 0.15C–0.40Si–1.51Mn (wt\%) steel.

It should be mentioned that the observation based on the quenched specimens is less reliable than \textit{in-situ} dilatometry measurement due to the difficulty in avoiding formation of ferrite during the quenching process, as we have discussed in the previous paper.\textsuperscript{29} In the present study, three different methods (determination of critical strain, microstructural observation and dilatometry) were used to detect the DT above $A_{E3}$ and all measurements come to the same conclusion that DT can indeed occur at temperature higher than $A_{E3}$ for the tested steel at a strain rate of 1 s$^{-1}$.

It is quite obvious that the raised $A_{E3}$ results from the increased stored energy due to deformation applied in the austenite. For the investigated steel, it requires the deformation stored energy of 35 J/mol to raise its $A_{E3}$ temperature from 1 108 to 1 133 K, as calculated by Thermo-Calc software. The deformation stored energy, $\Delta G_D$, can be approximately calculated from the stress by the equation\textsuperscript{40}:

$$\Delta G_D = C_2 \rho G b^2$$

where $C_2$ is a constant with the typical value of 0.5, and $G$, $b$ and $\rho$ are the shear modulus, Burgers vector and the dislocation density, respectively, which can be calculated from the following equations\textsuperscript{41}:

$$G = 1.45 \cdot 10^5 - 137 \cdot T + 3.48 \cdot 10^{-2} \cdot T^2, \text{ in MPa}$$ \hspace{1cm} (5)

$$b = \frac{\sqrt{2}}{2} \cdot (0.3260 \cdot (1 + 24.73 \cdot 10^{-6} \cdot (T - 1000))) \text{, in nm}$$

$$\rho = \left(\frac{\sigma}{\alpha G b}\right)^2$$ \hspace{1cm} (7)

where $T$ is the absolute temperature in Kelvin, $\sigma$ is the flow stress and $\alpha$ is a constant which can be approximately calculated by the following equation for C–Mn steel\textsuperscript{42}:

$$\alpha = -0.000628 \cdot T + 1.0693, \text{ in N/m}^2$$ \hspace{1cm} (8)

The deformation stored energy can thus be determined by Eqs. (4) through (8), where the flow stress is taken as the stress corresponding to the critical strain of DT according to the stress–strain curves. The calculated $\Delta G_D$ at 1 133 K is only about 22 J/mol, which is less than the required value (35 J/mol) to achieve orthoequilibrium between austenite and ferrite at this temperature. Moreover, it is not possible for the solute elements, such as Si and Mn, to diffuse within such a short deformation period (less than 1 s), and only the interstitials like carbon might be able to diffuse to some ex-

Fig. 9. Changes of the measured temperature and stroke with time during deformation test at 1 113 K (a) and at 1 133 K (b).

Fig. 10. Microstructures on the specimen immediately quenched after deformation at 1 123 K (a) and subject to 1.2 ks holding following deformation and then quenched (b).
tent during the dynamic transformation. Therefore, the required deformation energy for occurrence of dynamic transformation will be even more than 35 J/mol. The only reasonable explanation for this inconsistency is that the actual deformation stored energy does not distribute uniformly across the specimen; while the above-mentioned calculation assumes a homogeneous dislocation density throughout the deformed austenite. The exact microstructure in the deformed austenite, unfortunately, will be lost after quenching due to the austenite-to-martensite transformation. Hurley et al.\textsuperscript{11} and Adachi et al.\textsuperscript{37} have studied deformation substructure in the austenite by using a model alloy, Ni–30\%Fe, which has similar stack fault energy to the austenite of carbon steels. They concluded that the main deformation feature in the austenite is the formation of microshear bands with the increasing strain. Its formation involves the linking up of many small dislocation cells within a grain during inhomogeneous deformation, thus allowing flow to occur in a favorable direction under the resolved shear stress.\textsuperscript{12} Such microshear bands are preferential nucleation sites for recrystallization since they are regions of much higher stored energy than the average in bulk\textsuperscript{43}; similarly, they can be preferential nucleation sites for dynamic transformation as well. This, however, requires further efforts to identify the relation between such microshear bands in the austenite and dynamic ferrite transformation.

4. Conclusions

Post-dynamic transformation was investigated by dilatometry and microstructural observation in a low carbon steel. When the deformation temperature is between \( \Delta T_{\text{e}} \) and \( \Delta T_{\text{f}} \), an austenite-to-ferrite transformation could continue after DT during deformation, and its kinetics can be well described by the JMAK equation, but the value of the Avrami exponent \( n \) appears to decrease with increasing strain since larger strains lead to more ferrite dynamically formed during deformation, leading to an earlier impingement between the formed ferrite grains during the post-DT and consequently a reduced exponent of \( n \) due to the growth in less dimensions.

The occurrence of DT during deformation above \( \Delta T_{\text{e}} \) was also confirmed by combination of three experimental methods: the derivative of \( \theta-\sigma \) curves, optical microscope observation on quenched specimens and dilatometry measurement. The last one is most reliable since it is \textit{in-situ} measurement and the possible ferrite transformation during the quenching can be avoided. It is believed that a considerable rise in \( \Delta T_{\text{e}} \) temperature is due to deformation stored energy in the austenite. Particularly, the transformation temperature can be even raised more in regions with much higher stored energy, such as microshear bands, than the average in the bulk because the plastic deformation is often inhomogeneous.

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