Effects of plastic deformation on austenite transformation in Fe-1.93Mn-0.07Ni-1.96Cr-0.35Mo ultra-high strength steel during continuous cooling

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Abstract

Dynamic continuous cooling-transformation (D-CCT) diagram is an essential research method to predict mechanical properties of ultra-high strength steel (UHSS) before material processing. In this study, D-CCT diagrams of Fe-1.93Mn-0.07Ni-1.96Cr-0.35Mo at different cooling rates of 0.5–15 °C s⁻¹ were plotted by a combined method of dilatometry and metallography. Influences of deformation on microstructure, transformation behavior, and carbon diffusion were elucidated. Proeutectoid ferrite transformation behavior was discussed in detail based on strain induced nucleation kinetics. When the cooling rate increased above 1 °C s⁻¹, the initial austenite grains were divided into blocks by newly forming complex microstructures, which were mainly consisted of martensite (M), bainite (B), deformation induced ferrite (DIF) and retained austenite (RA). Moreover, the decrease of proeutectoid ferrite and the increase of granular bainite (GB) resulted in the strength increased linearly at the cooling rate of 0.25–2 °C s⁻¹. Increasing cooling rate has a remarkable effect on the tensile strength of the UHSS up to 1 °C s⁻¹. Further increase in the cooling rate from 1 to 15 °C s⁻¹, the results showed a relatively small effect on the intensity value. Besides, carbon diffusion from α-phase (face-centered-cubic microstructure) in DIF can enhance the stability of γ-phase (body-centered cubic microstructure) and delay the transformation of γ-phase to GB. Furthermore, attributed to the diffusion path of carbon decreases, the proeutectoid ferrite transformation was restrained, and γ-phases were transformed into GB as raising the cooling rate.

1. Introduction

The development of ultra-high-strength materials and alloys is one of the hotspots and difficulties of current materials research, and researchers have achieved remarkable results [1–7]. Steel manufacturers usually call steel materials with a tensile strength greater than 780 MPa as UHSS, which can reduce the steel consumption and improve the safety coefficient significantly that it is widely used in machinery, mining, building, military, aerospace and so on [8–10]. Compared to Static Continuous Cooling Transformation (S-CCT) of undeformed austenite, the D-CCT diagram is broadly applied to predict mechanical properties in hot working processes. Influences of deformed and subsequently cooling conditions, various microstructures, such as proeutectoid ferrite, deformation induced ferrite/strain induced ferrite, acicular ferrite, granular bainite, lath martensite, and residual austenite, may be obtained in the D-CCT diagram [11–13]. Consequently, it is meaningful to reveal the evolution mechanism for the regulation of microstructure and mechanical properties.

From the perspective of thermodynamics and grain refinement, deformation not only has a strong influence on the transformation of austenite to ferrite (F) but also is an essential way of thermo-mechanical control processing for achieving F refinement. Many researches [14–17] have been carried out to illustrate the effects of cooling, carbon content, deformed condition, and chemical composition on DIF transformation, and the
related theoretical system has been improved progressively. Tamura et al. [18] defined the transformation from deformation induced austenite to ferrite as deformation induced transformation (DIT), or strain induced/ enhanced transformation (SIT/SET). Meanwhile, in the corresponding temperature range, DIT can also induce alloy carbide precipitation [19, 20], and this characteristic has received widespread concern and discussion. Yang et al. [21, 22] believed that the transformation driving force of DIT is still the strain energy, which has a significant influence on the P phase transformation and occurs above Ac3. It is interesting to note that the occurrence of DIT contradicts the traditional thermodynamic viewpoint. Regarding the formation mechanism of DIT, the experimental results of Davenport et al. [23] showed that after the quenching of hyposteel, the core of the sample was P without pearlite in more than 90% volume content, which was not observed in the usual quenching and cooling process. Yada et al. [19] considered that when strain rate exceeds 250 s⁻¹, carbon diffusion had been suppressed, and DIF was insensitive to the strain rate. Whereas, at the lower strain rate zone, as the strain rate increases, the DIT increases. Therefore, carbon diffusion has influence on DIT, but the mechanism is controversial. Jonas et al. [24–28] and Petrov et al. [29] investigated the effects of strain on the strain transformation and proposed storing energy transformation model for the condition of low strains and dynamic transformation above the equilibrium transformation temperature.

In the present work, the thermal expansion method is used to determine the critical phase transformation temperatures of CCT. Influences of cooling rate on microstructure, carbon diffusion, and DIT transformation are analyzed. Furthermore, the strength and toughens mechanism of UHSS are put followed based on phase transformation.

2. Experimental materials and methods

2.1. Experimental materials
The experimental steel was smelted by a 150 kg vacuum induction furnace. Harmful elements of the test steel, such as S and P, were controlled strictly. The chemical compositions (wt%) of the steel were 0.23 C, 1.95Si, 1.93Mn, 0.007 P, 0.003 S, 0.07Ni, 1.96Cr, 0.35Mo, Nb + V + Ti ≪ 2.0. Then the sample was rolled into 10 mm thick steel sheet within the temperature range of 1100 °C–1180 °C by φ450 hot rolling mill, and the finished sheet was air cooled. Then φ 4 mm × 10 mm phase transformation instrument sample and φ 8 mm × 15 mm thermal simulation sample were prepared. The metallographic specimens were etched by 4% nitric (Vol%) after being grinded and polished. The microstructure was observed by Leica 2500 M (Leica, Wetzlall, Germany) microscope. The microstructure of examined steel is mainly composed of M and B, as shown in figure 1(a).

Tensile test specimens with a size of φ6 mm (d₀), 50 mm length of the reduced section (L₀) and a gauge length of 40 mm (Lₘ) were machined according to the Chinese standard GB/T 228–2010. The clamping sections at both ends of the specimens were processed into a thread of M10.0. Tensile properties were tested according to the Chinese standard GB/T 228.1–2010 by WAW-1000 (SUST Electric Equipment Co., Ltd, Zhuhai, China) universal tensile testing machine to determine the engineering stress. Vickers hardness test was performed according to the Chinese standard GB/T 4340.1–2009 using a KB3000BVRZ-SA Vickers hardness tester (Leica, Wetzlall, Germany) with a loading force of 9.8 kg and holding 10 s. The mechanical properties, including tensile and hardness of the tested steel, are presented in figure 1(b), which displayed that values of Yield Strength (YS), Tensile Strength (TS), Elongation (A), and Vickers Hardness (HV) are 1260 MPa, 1570 MPa, 16%, and 395 N mm⁻²⁻², respectively.
2.2. Determination of critical transformation temperature
Before the determination of the critical phase transformation temperature in the continuous cooling process of the experimental steel, the fitted empirical formulas \[30-33\] were used to estimate the temperatures of \(A_{c1}\) and \(A_{c3}\) for predicting experimental parameters of heat treatment. Then, the critical austenite transformation temperatures \(A_{c1}\) and \(A_{c3}\) were determined by dilatometry using Formastor-FII (Fuji Electronic Industrial Co., Ltd, Saitama, Japan) automatic phase transformation instrument.

2.3. Determination of D-CCT diagrams
The determination experiment of D-CCT diagrams was carried out on the MMS-300 thermomechanical simulation system (Northeastern University, Shenyang, China). The experimental scheme, according to the parameters of the sheet, strip and bar steel in the rolling process, such as the hot deformed temperature, rolling speed, and rolling reduction ratio is shown in figure 2. Firstly, the sample was heated with 10 °C s \(^{-1}\) to 1200 °C and holding 600 s to obtain the complete austenitic microstructure. Secondly, the sample cooled with 10 °C s \(^{-1}\) to 920 °C and holding 10 s. Then, a single pass compression deformation with a true strain of 0.4 was performed at a strain rate of 0.5 s \(^{-1}\) to simulate the hot rolling process in the factory. Finally, the deformed samples were cooled to room temperature (about 20 °C) at a controlled cooling rate of 0.5 °C s \(^{-1}\), 1 °C s \(^{-1}\), 2 °C s \(^{-1}\), 5 °C/s, 10 °C s \(^{-1}\), 15 °C s \(^{-1}\), respectively. Furthermore, dilatometry was used to determine the phase transformation temperature in the D-CCT process.

2.4. Microstructural characterization
Dilatometer specimens were cut along the axis by wire spark cutting machine and etched with 4% (Vol%) nitric acid alcohol after being grinded and polished. Then the morphology of the tested sample was obtained by Leica DM 2500 M (Leica, Wetzlall, Germany) microscope.

3. Results

3.1. Critical transformation temperature
The fitted empirical formulas (1) and (2) are usually applied to estimate the phase transformation temperatures of \(A_{c1}\) and \(A_{c3}\) [30–33]:

\[
A_{c3} = 910 - 203C + 44.7Si - 30Mn - 20Cu - 15.2Ni - 11Cr + 31.5Mo + 104V + 400Ti + 400Al = 903 \ ^\circ C
\]

\[
A_{c1} = 751 - 16.3C + 34.1Si - 27.5Mn - 5.5Cu - 15.9Ni + 12.7Cr + 3.4Mo = 784 \ ^\circ C
\]

The calculated transformation temperature provides guidance for selecting the austenite solution temperature. Based on calculation results, the dilatometry experiment was carried out at a heating rate of 0.05 °C s \(^{-1}\) in the range of 700 and 960 °C. The difference in expansion and shrinkage coefficients of bcc (\(\alpha\)-phase) and fcc (\(\gamma\)-phase) destroys the linear relationship between the expansion and temperature when the phase transformed from \(\gamma\)-phase to \(\alpha\)-phase. This feature is well reflected in the dilatometric curve of the examined steel, as shown in figure 3. Furthermore, the critical transformation temperatures \(A_{c1} = 798 \ ^\circ C\) and \(A_{c3} = 901 \ ^\circ C\) were determined by using the tangent method.
3.2. D-CCT diagram and microstructure

D-CCT diagrams obtained by the dilatometry method combined with microstructure observation are shown in figure 4. Figure 5 displays the microstructures of deformed austenite at different cooling rates. It can be seen from figure 4 that in a wide cooling rate range of 0.5 – 20 °C s⁻¹, there is DIF along the prior austenite grain boundary and contains a large amount of bainite. At the low cooling rate (0.5 – 1 °C s⁻¹), the preponderant polyphase microstructures are proeutectoid ferrite and bainite, which transformed into bainite and lath martensite between 2 and 20 °C s⁻¹. Simultaneously, when the cooling rate in the range of 1 – 20 °C s⁻¹, raising the cooling rate leads to the decrease of the bainite transformation temperature. In the lower cooling rate, decreasing the cooling rate is conducive to the formation of proeutectoid ferrite. Obviously, the carbon content in the supercooled austenite decreases inevitably because of the formation of the partial proeutectoid ferrite, which has not diffused to austenite, and it is beneficial to the bainite transformation.

Continue to increase the cooling rate, since the proeutectoid ferrite is suppressed, the supercooled austenite nearly has the same carbon content, and the starting temperature of bainite transformation is mainly dependent on the cooling rate. As can be seen from figure 4, the finish transformation temperature of bainite is marked by the gradual increasing tendency at the cooling rate higher than 2 °C s⁻¹. In detail, when the cooling rate increases from 2 to 5 °C/s, the starting transformation temperature of bainite decreases. The higher cooling rate can provide a larger driving force for the transformation, which suppresses the formation of proeutectoid ferrite, increases the efficient nucleation sites of bainite, and is beneficial for the generation of lath bainite. Eventually,
the finish transformation temperature is lowered. When the cooling rate reaches to 5–20 °C s\(^{-1}\), both the start and finish temperatures increase. This characteristic proves that deformation can cause more dislocation defects and provide more nucleation points. At the same time, both proeutectoid ferrite and granular bainite are suppressed under the higher cooling rate. Besides, the diffusion time of carbon is shortened, lath bainite and
martensite are formed in a shorter transformation time. As a consequence, the start and finish temperatures increase.

4. Discussion

4.1. Effects of deformation on D-CCT diagram

Based on the analysis of the deformed austenite transformation behavior for the examined steel, it is noteworthy that the deformation has a significant influence on the DIF under the same heating and cooling conditions. Dong Han et al. [34] established the critical nucleation energy of F in different conditions.

Critical nucleation energy of proeutectoid ferrite in the undeformed $\gamma$-phase can be calculated as follows [34]:

$$\Delta G_{undeform} = \frac{4\eta^2\sigma^3}{27(\Delta G_A + U_A)^2}$$  \hspace{1cm} (3)

While, the following expression can be used to describe the critical nucleation energy of DIF in the deformed $\gamma$-phase [34]:

$$\Delta G_{deform} = \frac{4\eta^2\sigma^3}{27(\Delta G_A - \Delta G_D + U_A)^2}$$ \hspace{1cm} (4)

Where, $\Delta G_A$ is the free enthalpy change value of each atom in the crystal, and value of $\Delta G_A$ is always negative; $U_A$ is the strain energy of each atom in the crystal; $\sigma$ is the surface energy; $\eta$ is the shape factor; $\Delta G_D$ is increment value of free enthalpy of atom in the crystal defect.

The ratio for the nucleation of deformation to non-deformation in the process of $\gamma \rightarrow \alpha$ transformation [34]:

$$\frac{I_{deform}}{I_{undeform}} = \exp \left(\frac{\Delta G_{undeform}^* - \Delta G_{deform}^*}{kT}\right)$$  \hspace{1cm} (5)

Where, $k$ is the Boltzmann constant.

It can be seen from the equations (3)–(5) that the deformation can greatly improve the nucleation rate in the process of $\gamma \rightarrow \alpha$ transformation, and it can promote more than ten orders of magnitude. The increase of the nucleation rate is also attributable to the deformation, which causes a decrease in the $\Delta G_{deform}$ of the DIF and the increase of the dislocation defects. In conclusion, it is difficult to suppress the formation of the DIF in high temperatures (above $A_g$ temperature).

It can be seen in figure 5, a tiny amount of F still appears when the cooling rate reaches 2 °C s$^{-1}$ in the D-CCT diagram. However, our previous research showed that F content in S-CCT was very low, even at a low cooling rate of 1 °C s$^{-1}$ [35]. It can be speculated that the deformation of austenite in the non-recrystallized zone can promote the precipitation of DIF and inhibit the bainite transformation.

4.2. Effects of deformation on austenite start transformation temperature

Figure 6 shows the change of austenite start transformation temperature ($A_s$) with a cooling rate in the S-CCT diagram and the D-CCT diagram at 900 °C. Compared with (a) and (d) in figure 6, it can be seen that deformation makes the bainite transformation temperature rise, which indicates that deformation can promote the bainite transformation. The reason for the promotion is that the deformation increases the density of defects such as dislocations and deformation bands in non-recrystallized austenite, which effectively increases the nucleation rate of $\gamma \rightarrow \alpha$ phase transformation and leads to the higher phase transformation temperature. Khlestov et al. [36] considered that the deformation would inhibit the bainite phase transformation at a lower temperature (250–350°C), but accelerate this transformation at higher temperatures (400–500 °C). The experimental result is consistent with Khlestov’s conclusion. It should be pointed out that the effect of deformation on bainite phase transformation is also relevant to precipitation of proeutectoid ferrite before the bainite phase transformation. If a large amount of proeutectoid ferrite appears in the microstructure of UHSS, deformation can inhibit the bainite phase transformation. As can be seen from figures 5 and 6, a small amount of proeutectoid ferrite appears at a lower cooling rate after deformation, and this very few proeutectoid ferrites has no significant effect on the phase transformation process of bainite.

4.3. Effects of deformation on carbon diffusion

Du et al. [37, 38] considered that proeutectoid ferrite precipitated from austenite was a diffusion type transformation, and deformation induced ferrite transformation (DIFT) is the result of the coincidence of deformation temperature and the increase of transformation caused by deformation when the strain rate was 1–10 s$^{-1}$. Therefore, DIFT is essentially consistent with the proeutectoid ferrite precipitated from austenite. Liu
et al. [39, 40] found that the carbon concentration in DIF was close to that in the original austenite, showing an apparent phenomenon of carbon supersaturation. Consequently, Liu et al. [39, 40] speculated that there was no obvious diffusion of carbon from DIF to austenite, and the microstructure (tetragonal martensite) obtained by DIFT was in an extremely unstable state because of the supersaturated carbon.

As has been described previously, increasing the driving force of the phase transformation would lead to the formation of proeutectoid ferrite during the cooling process, at which time the higher carbon in the DIFT would diffuse to the proeutectoid ferrite and γ-phase, and the DIFT would transfer to the proeutectoid ferrite. Therefore, based on the thermodynamic analysis in the DIFT process, the storage energy generated by deformation cannot be released entirely in a short time after deformation, and a part of the stored energy can be retained in the deformed structure. At a suitable cooling rate, this stored energy is transformed into a part of the free energy in the process of phase transformation. Finally, the stored energy increases the phase transformation driving forces of proeutectoid ferrite and bainite, so the $\gamma \rightarrow \alpha$ transformation temperature increases on the whole (Region I and Region II in figure 6).

At that same time, however, it was found that when the cooling rate increased from 0.5 °C s$^{-1}$ to 1 °C s$^{-1}$, the $A_{s3}$ temperature decreased significantly (region III). This phenomenon may be partially due to the increase of the cooling rate, which can decrease the diffusion time of carbon at high temperature, lessen the content of carbon in proeutectoid ferrite to γ phase, reduce the stability of γ-phase, and increase the $\gamma \rightarrow \alpha$ transformation temperature. In deformed austenite, increasing the cooling rate from 1 °C s$^{-1}$ to 2 °C s$^{-1}$ results in the increase of $A_{s3}$ temperature. This phenomenon not appears in the undeformed austenite and is attributed to the diffusion of supersaturated carbon in strain induced ferrite. Therefore, the deformation of the non-recrystallization zone at 900 °C promotes the phase transformation of $\gamma \rightarrow \alpha$, and the phase transformation shifts to high temperature. At the same time, it is not difficult to find that the strain can inhibit the phase transformation of $\gamma \rightarrow$ GB. Strain induced carbon diffusion of $\alpha \rightarrow \gamma$ promotes γ-phase stability and delays the transformation of $\gamma \rightarrow$ GB. Increasing the cooling rate can further decrease the diffusion path of carbon, restrain the proeutectoid ferrite

Figure 6. Relationship between the austenite transformation temperature and the cooling rate at 900 °C: (a) Undeformed austenite transformation behavior [35]; (b) Vickers hardness (HV) of the undeformed austenite [35]; (c) Start transformation temperature ($A_{s3}$) of the undeformed and deformed austenite; (d) Effects of deformation on the microstructure transformation compare with undeformed and deformed austenite.
phase transformation and make $\gamma$-phase transformed into GB. When the cooling rate higher than 5 °C/s, the mechanism of diffusion phase transformation is similar to that of the S-CCT diagram.

5. Conclusions

In order to study the D-CCT diagram of UHSS, the critical temperature of austenitic transformation was measured, and the behavior of deformed austenitic continuous cooling transformation was discussed. The influences of cooling rate on the microstructure transformation and carbide precipitation were analyzed in detail. Also, the transformation behavior of proeutectoid ferrite during $\gamma \rightarrow \alpha$ transformation was elucidated. The main conclusions are summarized as follows:

1. The critical temperatures $A_{c1}$ and $A_{c3}$ are 746 °C and 868 °C, respectively. When the cooling rate increased above 1 °C s$^{-1}$, austenite grains were divided into blocks by the formed complex microstructures of M, B, and RA, et al.

2. When the cooling rate in the range of 0.25–2 °C s$^{-1}$, PF has a significant influence on the hardness. With the decrease of PF content and the increase of GB content, the strength increases linearly. The tensile strength can be controlled in a specific range with the cooling rate at 0.25–2 °C s$^{-1}$. The cooling rate over 10 °C s$^{-1}$ has little effect on the tensile strength.

3. The carbon diffusion from $\alpha$-phase in DIF to $\gamma$-phase enhances the stability of $\gamma$-phase and delays the transformation of $\gamma \rightarrow$ GB. Increase the cooling rate can decrease the diffusion path of carbon, restrain the PF phase transformation, and make $\gamma$-phase transformed into GB.

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