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Continuous Cooling Transformation of Under-Cooled Austenite of SXQ500/550DZ35 Hydropower Steel

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Abstract: The expansion curves of the continuous cooling transformation of undercooled austenite of SXQ500/550DZ35 hydropower steel at different heating temperatures and cooling rates were measured by use of a DIL805A dilatometer. Combined with metallography and Vickers hardness measurement, the continuous cooling transformation diagrams (CCT) of the studied steel under two different states were determined. The results show that in the first group of tests, after the hot-rolled specimens were austenitized at 920 °C, when the cooling rate was below 1 °C·s⁻¹, the microstructure was composed of ferrite (F), pearlite (P) and bainite (B). With the cooling rates between 1 °C·s⁻¹ and 5 °C·s⁻¹, the microstructure was mainly bainite, and martensite (M) formed as the cooling rate reached 5 °C·s⁻¹. When the cooling rate was up to 10 °C·s⁻¹, the microstructure was completely martensite and the hardness value increased significantly. In the second group of tests, after the hot-rolled specimens were quenched at 920 °C and then heated at an intercritical temperature of 830 °C, in comparison with the first group of tests, and except for additional undissolved ferrites in each cooling rate range, the other microstructure types were basically the same. Due to the existence of undissolved ferrite, the microstructures of the specimens heated at intercritical temperatures were much finer, and the toughness values at low temperatures were better.

Keywords: SXQ500/550DZ35 hydroelectric steel; CCT diagrams; intercritical annealing; granular bainite

1. Introduction

SXQ500/550DZ35 hydropower steel plate is a kind of low-carbon, low-alloy, high-strength steel with quenched and tempered processes, which is usually designed to be used for the seat ring of hydropower equipment. The microstructure of this steel plate is designed to be low bainite, which requires high strength, good weldability, and high low-temperature toughness. Therefore, a suitable heat treatment process to produce high strength and toughness with ultra-thickness steel plates is needed [1,2]. At present, the traditional heat treatment process for ultra-thickness steel plate is still the quenching and tempering processes, which can obtain the required strength, but the obtained low temperature toughness is usually not high enough and cannot meet the user’s requirements [3–6].

In recent years, some researcher suggested a new type of heat treatment process with intercritical quenching and tempering [7–11]. The intercritical heat treatment process comprises hypoeutectoid steel with an equilibrium or non-equilibrium original microstructure being heated to intercritical temperature zone and held for some time, then quenched or isothermal hardened or normalized. The intercritical heat treatment process requires a definite original microstructure of steels, which is provided by a preparatory heat treatment process, such as a complete quenching or quenching and tempering process [12–15]. It was shown that the microstructure resulting from the intercritical process can be effectively refined with a few ferrite grains formed, resulting in a good match between the strength and toughness of steels, which is beneficial to industrial production. Therefore, this work...
intends to design the combination of a low-carbon bainite microstructure and intercritical annealing, which will adopt a ductile phase and refined microstructure to enhance the strength and toughness of steels.

A continuous cooling transformation diagram of the undercooled austenite of steels (CCT) can quite intuitively show the microstructure type, transformation time and transformed phase amount, which not only provides a theoretical basis for the suitable heat treatment process of steels, but also plays an important role in studies about the development of new grades of steels and new heat treatment processes.

Research and development of ultra-thickness steel plates SXQ500/550DZ35 have not yet been completed and few studies about phase transformation thermodynamics and kinetics have been reported so far. Therefore, this work will study the CCT diagrams of single-phase, under-cooled austenite and the intercritical zone, deciding the microstructure and properties of the experimental steels with different cooling rates, then providing suitable technology parameters for intercritical annealing of SXQ500/550DZ35 steels.

2. Materials and Methods

2.1. Experimental Material

The materials were obtained from a steel company that produced SXQ500/550DZ35 hot-rolled ultra-thickness steel plates, whose composition is shown in Table 1. The steel plate thickness is 260–300 mm.

| C     | Si  | Mn  | P   | S   | V   | Ti  | Als | O   |
|-------|-----|-----|-----|-----|-----|-----|-----|-----|
| 0.11  | 0.12| 1.18| 0.009| 0.001| 0.042| 0.004| 0.019| 0.0015|
| Cr    | Mo  | Nb  | Ni  | Cu  | B   | N   | As  | -   |
| 0.506 | 0.386| 0.035| 1.380| 0.020| 0.0013| 0.0058| 0.017| -   |

2.2. Experimental Method

The experimental equipment was thermal dilatometer DIL805A.

The specific experimental procedures were as follows:

(1) Determination of austenitization temperature.

Based on Thermo-Calc thermodynamic calculation software (2017b, Thermo-Calc Software, Stockholm, Sweden), the precipitate type, precipitation temperature and precipitate maximum amount at equilibrium for the experimental steel are determined and the solid solution rule of microalloying elements Nb, Ti, V in austenite are thermodynamically calculated [16]. Simultaneously, the grain sizes at different austenitizing temperatures are observed, and then the suitable austenitizing temperature are determined. The heat treatment process in this work is as in Figure 1.

![Figure 1. Heat treatment process.](image)

(2) Determination of critical points.

Hot rolled specimens were heated to 400 °C at a rate of 10 °C.s⁻¹, and then to 920 °C with a rate of 0.055 °C.s⁻¹. After being held for 22 min, the specimens were cooled to room
temperature with a rate of 0.055 °C·s\(^{-1}\), and the critical transition points \(A_{c1}\) and \(A_{c3}\) were measured during the heating process; the specific procedure is shown in Figure 2 [17,18].

![Figure 2. Heating process diagram.](image)

According to the YB/T5127-1993 ‘measurement method for critical points of steels (thermal expansion method)’ [19], the thermal expansion method is used to measure the static critical points of steels. The specific method is that tangent method is used to take the separation point between the extension line of the linear part of the thermal expansion curve and the curve part as the critical point.

(3) The first group of specimens was heated to 920 °C with a rate of 10 °C·s\(^{-1}\) in thermal dilatometer DIL805A and after being held for 22 min, the specimens were cooled to room temperature with rates of 0.05, 0.1, 0.6, 1, 2, 3, 5, 10, 35, 50 °C·s\(^{-1}\), respectively, as shown in Figure 3a. The second group of specimens was heated to 920 °C and after being held for 22 min, the specimens were quenched; then, they were again heated to intercritical temperature 830 °C and after being held for 22 min, they were cooled to room temperature with rates of 0.05, 0.1, 0.6, 1, 2, 3, 5, 10, 35, 50 °C·s\(^{-1}\), respectively, as shown in Figure 3b.

![Figure 3. Process diagram (a) first group test; (b) second group test.](image)

(4) The specimens size required for this test was φ4 mm × 10 mm. After experiment on a thermal dilatometer, cut radially on the dilatometer, the two groups of specimens that had undergone different heat treatment processes were made to be metallographic specimens. After being ground, polished and etched 5 s with 4% nital, the microstructures of the specimens were observed with an optical microscope and scanning electron microscopy, and the hardness values of the microstructures for the specimens were measured with 430SH Vickers hardness tester (load was 0.5 kg).

3. Experimental Results and Discussion

3.1. Determination of Austenitization Temperature

The calculation results are shown in Figure 4 by use of Thermo-Calc thermodynamic software [16], from which it can be concluded that the critical points \(A_{c1}\) and \(A_{c3}\) are 705 °C and 834 °C, respectively, for the studied steel. In the temperature range from 800 °C to 1400 °C, the solid solubility values of the microalloying elements V, Nb and Ti in austenite changed with the temperature, as seen in Figure 5.
3. Experimental Results and Discussion

3.1. Determination of Austenitization Temperature

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The austenite (A) grain morphologies of the hot-rolled specimens are shown in Figure 6 when heated at different temperatures. It is shown that with increasing temperature, the austenite size increases continuously. According to statistics, the relationship curve between the average grain diameter and heating temperature is shown in Figure 7. It is shown that the austenite grains start to grow quickly at 970 °C, and the coarsening temperature of the austenite grains of the experimental steel is 1020 °C. With consideration of the complete solution of microalloying elements and austenite grain size, 920 °C was selected to be the austenizing temperature for the measurement of the CCT diagram for single-phase austenite. After intercritical annealing, the average grain diameter decreases from 19.67 $\mu$m to 9~11 $\mu$m. The grain is refined obviously after intercritical annealing. The grain size growth curve shows that the grain grows rapidly at 840 °C. Therefore, 830 °C was selected to be the heating temperature for the measurement of CCT for the intercritical annealing.

Figure 4. Equilibrium precipitates in SXQ500/550DZ35 steel.

Figure 5. The composition of austenitic phase elements in the experimental steel.

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830 °C was selected to be the heating temperature for the measurement of CCT for the intercritical annealing.

**Figure 6.** Austenite grain morphology of the hot-rolled specimens at different heating temperatures (a) 850 °C; (b) 870 °C; (c) 920 °C; (d) 970 °C; (e) 1020 °C; (f) 1070 °C.
3.2. Determination of Critical Points

The critical points $A_{e1} = 744 \degree C$, $A_{e3} = 847 \degree C$ of the experimental steel were obtained through experiments. The thermodynamic calculation results of the equilibrium critical points were $A_{e1} = 705 \degree C$, $A_{e3} = 834 \degree C$. The difference between the thermodynamic calculation results and the experimental values of the critical points is not large, which shows that the thermodynamic results are believable and can be taken as reference.

3.3. Microstructure and Hardness of the Experimental Steel at Different Cooling Rates

In order to better decide the transformed products of under-cooled austenite of the experimental steel, it is necessary that the microstructures at different cooling rates of the experimental steel are observed and the microhardness measured.

For the first group of specimens, for the rolled specimens heated to 920 °C for austenitization, the microstructures at different cooling rates are shown in Figures 8 and 9. With a cooling rate of 0.1 °C·s$^{-1}$ or below, since the carbon and iron atoms had enough time to diffuse in undercooled austenite and through the interface of $\gamma/\alpha$, ferrite and pearlite microstructure formed. With decreasing temperature, under-cooled austenite transformed to bainite with semi-diffusion mechanism, which included few granular bainite (GB) of lumpy ferrite and islands with carbon-rich austenite, in the medium temperature range. The average hardness for these specimens was relatively low [20–23]. With the cooling rate increasing to 0.6 °C·s$^{-1}$, the granular bainite amount increased gradually in the medium temperature range, and the average hardness values of these specimens increased. With a cooling rate of 1 °C·s$^{-1}$, lath bainite (LB) formed, in which ferrite and carbide crisscrossed in a lath bundle and were distributed approximately parallel to each other at long intervals. With the cooling rate ranging from 2 °C·s$^{-1}$ to 5 °C·s$^{-1}$, more lath bainite formed, and some martensite formed at a cooling rate of 5 °C·s$^{-1}$. With increasing the cooling rate to 10 °C·s$^{-1}$, the lath bainite disappeared in the microstructure and martensite took the most part; the hardness value apparently increased. When the cooling rate reached 35 °C·s$^{-1}$ after austenitization of the specimen, the carbon and iron atoms had insufficient time to diffuse, due to the large cooling rate; thus, martensite transformation occurred with shear and no composition diffusion mechanism in the specimens. Since the carbon content was low in the experimental steel and the $M_s$ point was relatively high for this steel, the lath martensite formed with the slipping mechanism had high density dislocation [24].

For the second group of specimens, after the specimens were heated to 920 °C and held for 22 min, the specimens were quenched, and then reheated to the intercritical temperature of 830 °C, held for 22 min, and cooled to room temperature with different cooling rates; the microstructures are shown in Figures 10–12. It is shown that there occurred ferrite, pearlite, bainite and martensite transformations with different cooling rates. Compared to the first group of specimens, there always existed undissolved ferrite in each cooling rate range for this group of specimens. When the cooling rate reached 1 °C·s$^{-1}$ or above, no more eutectoid ferrite formed, and the ferrite in the microstructure was undissolved [25,26].
Therefore, it is inferred that the low temperature toughness and strength of the specimen with intercritical annealing is further enhanced.

**Figure 8.** Metallographic microstructure of the rolled specimens at different cooling rates (a) original microstructure; (b) 0.05 °C·s\(^{-1}\); (c) 0.1 °C·s\(^{-1}\); (d) 0.6 °C·s\(^{-1}\); (e) 1 °C·s\(^{-1}\); (f) 2 °C·s\(^{-1}\); (g) 3 °C·s\(^{-1}\); (h) 5 °C·s\(^{-1}\); (i) 10 °C·s\(^{-1}\); (j) 35 °C·s\(^{-1}\); (k) 50 °C·s\(^{-1}\).

**Figure 9.** SEM morphology of the rolled specimens at different cooling rates (a) original microstructure; (b) 0.05 °C·s\(^{-1}\); (c) 0.1 °C·s\(^{-1}\); (d) 2 °C·s\(^{-1}\); (e) 10 °C·s\(^{-1}\); (f) 50 °C·s\(^{-1}\).
Figure 10. Microstructures of the quenched specimens at different cooling rates (a) original microstructure; (b) 0.05 °C·s$^{-1}$; (c) 0.1 °C·s$^{-1}$; (d) 0.6 °C·s$^{-1}$; (e) 1 °C·s$^{-1}$; (f) 2 °C·s$^{-1}$; (g) 3 °C·s$^{-1}$; (h) 5 °C·s$^{-1}$; (i) 10 °C·s$^{-1}$; (j) 35 °C·s$^{-1}$; (k) 50 °C·s$^{-1}$.

Figure 11. SEM morphology of the quenched specimens at different cooling rates (a) original microstructure; (b) 0.05 °C·s$^{-1}$; (c) 0.1 °C·s$^{-1}$; (d) 2 °C·s$^{-1}$; (e) 10 °C·s$^{-1}$; (f) 50 °C·s$^{-1}$.
Table 2. Microstructure types and hardness values of the specimens under different cooling rates.

| Cold Speed/°C·s⁻¹ | First Group | Hardness | Second Group | Hardness |
|------------------|-------------|----------|--------------|----------|
| a                 | original microstructure | F + GB + P | 244 | GB + LB | 240 |
| b                 | 0.05        | F + P + GB | 227 | F + P + GB | 232 |
| c                 | 0.1         | GB + F + P | 233 | GB + F + P | 262 |
| d                 | 0.6         | GB + F + LB_{less} | 256 | GB + F | 305 |
| e                 | 1           | GB + LB | 278 | GB + F | 307 |
| f                 | 2           | GB + LB | 309 | GB + LB + F | 325 |
| g                 | 3           | GB + LB | 328 | GB + LB + F | 324 |
| h                 | 5           | LB + M_{less} | 345 | LB + M_{less} + F | 344 |
| i                 | 10          | M + LB_{less} | 387 | M + LB_{less} + F | 405 |
| j                 | 35          | M + A' | 448 | M + A' + F | 456 |
| k                 | 50          | M + A' | 422 | M + A' + F | 454 |

Note: Fs, Ps, Bs, Ms: start temperature; Ff, Pf, Bf, Mf: finish temperature.

Figure 12. Hardness of the rolled and quenched specimens at different cooling rates.

The statistical results are shown in Table 2 of the microstructure types and hardness values for two groups of specimens. It is shown that two states of the specimens underwent ferrite, pearlite, bainite and martensite transformations during the continuous cooling process. The observation results of the microstructures show, for the two types of specimens with the same cooling rate, that the microstructure of the specimen with intercritical annealing is much finer and more homogeneous since undissolved ferrite was dispersively distributed with a needle-like or granular phase, which was able to hinder the grain boundary movement and grain growth of austenite, thereby effectively refining the austenite grains. The undissolved ferrite with low hardness and good ductility can prevent stress concentration and crack propagation, and therefore, it can enhance the low temperature toughness of steels. The hardness change trends of the two types of the specimens were compared, as seen in Figure 12. With increasing the cooling rate, the hardness values gradually increased and with the same cooling rate, the specimen with intercritical annealing had relatively larger hardness value. Generally, there exists a positive relationship between hardness and strength. After intercritical annealing, the interlacing microstructure of bainite and ferrite in steel was similar to that of “fiber reinforced composites” [27]. Bainite on grain boundary plays an important role in strengthening the grain boundary and improving its properties. The interface of ferrite and bainite phase is highly common; there is no formation of brittle carbides on the interface, and it is not easy to produce local stress concentration. The soft ferrite phase can reduce the yield ratio, but also prevents crack propagation and further improves the impact toughness. A large number of ferrite/bainite grain boundaries can effectively prevent crack propagation in steel. With increasing the cooling rate, the carbon atoms’ diffusion was not full; therefore, the solid solution strengthening of carbon atoms became stronger.

Therefore, it is inferred that the low temperature toughness and strength of the specimen with intercritical annealing is further enhanced.
3.4. Drawing of CCT Curve of the Experimental Steel

Based on the thermal expansion curve, the tangent method was adopted to the characteristic temperatures of under-cooled austenite at different cooling rates as shown in Tables 3 and 4. The characteristic temperatures were added to the corresponding cooling rate curve for different specimens, connecting the transition start points and final points with the same nature, marking out points \(A_{c1}, A_{c3}\) and \(M_s\). The CCT diagram of the experimental steel can be obtained by indicating the microstructure name in the corresponding phase transition zone, as shown in Figures 13 and 14. At the end of each cooling rate curve, the corresponding hardness value and cooling rate are marked.

**Table 3.** The phase transition points of the specimens after austenitizing at 920 °C.

| Cold Speed/°C·s\(^{-1}\) | \(F_s/°C\) | \(F_f/°C\) | \(P_s/°C\) | \(P_f/°C\) | \(B_s/°C\) | \(B_f/°C\) | \(M_s/°C\) | \(M_f/°C\) |
|---------------------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
| 0.05                      | 721         | -           | 623         | -           | 535         | 390         | -           | -           |
| 0.1                       | 699         | -           | 608         | -           | 552         | 404         | -           | -           |
| 0.6                       | -           | -           | -           | -           | 571         | 402         | -           | -           |
| 1                         | -           | -           | -           | -           | 574         | 403         | -           | -           |
| 2                         | -           | -           | -           | -           | 556         | 386         | -           | -           |
| 3                         | -           | -           | -           | -           | 546         | 370         | -           | -           |
| 5                         | -           | -           | -           | -           | 530         | -           | -           | 337         |
| 10                        | -           | -           | -           | -           | 512         | -           | -           | 289         |
| 35                        | -           | -           | -           | -           | -           | -           | 435         | 267         |
| 50                        | -           | -           | -           | -           | -           | -           | 413         | 265         |

Note: \(F_s, P_s, B_s, M_s\): start temperature; \(F_f, P_f, B_f, M_f\): finish temperature.

**Table 4.** The phase transition points of the different specimens heated at 830 °C.

| Cold Speed/°C·s\(^{-1}\) | \(F_s/°C\) | \(F_f/°C\) | \(P_s/°C\) | \(P_f/°C\) | \(B_s/°C\) | \(B_f/°C\) | \(M_s/°C\) | \(M_f/°C\) |
|---------------------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
| 0.05                      | 707         | -           | 631         | -           | 524         | 365         | -           | -           |
| 0.1                       | 695         | -           | 628         | -           | 540         | 382         | -           | -           |
| 0.6                       | 691         | -           | 624         | -           | 538         | 370         | -           | -           |
| 1                         | -           | -           | -           | -           | 571         | 390         | -           | -           |
| 2                         | -           | -           | -           | -           | 559         | 371         | -           | -           |
| 3                         | -           | -           | -           | -           | 551         | 336         | -           | -           |
| 5                         | -           | -           | -           | -           | 539         | 332         | -           | -           |
| 10                        | -           | -           | -           | -           | 497         | -           | -           | 302         |
| 35                        | -           | -           | -           | -           | -           | -           | 421         | 277         |
| 50                        | -           | -           | -           | -           | -           | -           | 407         | 265         |

**Figure 13.** CCT curve of single-phase region.
SXQ500/550D hydropower steel is a low-carbon, bainite, ultra-thickness plate. The study about the CCT curve of single-phase austenite showed that when the cooling rate is controlled in the range of 1 °C·s\(^{-1}\)−5 °C·s\(^{-1}\), the bainite microstructure can be obtained. Based on the study about the CCT curve of the intercritical process, it is shown that after the specimen was quenched at 920 °C and underwent the intercritical process at 830 °C, the microstructure of bainite and undissolved ferrite could be obtained, which were well matched in strength and toughness, with the cooling rate controlled at 1 °C·s\(^{-1}\)−5 °C·s\(^{-1}\).

Since undissolved ferrites distribute dispersively in an acicular or granular state, they can prevent stress concentration and crack propagation. Therefore, at the same cooling rate, the microstructure of the specimen, being quenched at 920 °C and undergoing the intercritical process at 830 °C, was much refined, and the strength and toughness were further enhanced.

4. Conclusions

Through the above experiments and analysis, the following conclusions are drawn:

(1) The average size of austenite grain increases with the increase in heating temperature. The grain coarsening temperature is 1020 °C, and it has a tendency to grow rapidly at 970 °C. The main reason for grain coarsening is that the carbon and nitrogen compounds of Nb and Ti begin to dissolve at 1020 °C, leading to a significant reduction in the number of particles in the second phase, weakening of the pinning effect, and even failure. Some grain boundaries break through the shackles and move and grow preferentially. When the temperature reaches 1070 °C, the grain size still grows but tends to homogenize, and the mixed crystal effect disappears.

(2) The critical transition temperatures \(A_{c1}\) and \(A_{c3}\) of the experimental steel are 744 °C and 847 °C, respectively.

(3) The CCT diagrams of the specimens in two states show that there are four transition zones: ferrite, pearlite, bainite and martensite. The CCT diagrams of single-phase under-cooled austenite shows that with the cooling rate below 1 °C·s\(^{-1}\), the microstructure consisted of ferrite, pearlite and bainite; with the cooling rate controlled at 2−5 °C·s\(^{-1}\), the microstructure was mainly composed of bainite; with the cooling rate at 5 °C·s\(^{-1}\), martensite started to occur in the microstructure; with the cooling rate at 10 °C·s\(^{-1}\), martensite was the main phase in the microstructure, with the hardness value apparently increased; with the cooling rate at 35 °C·s\(^{-1}\), the microstructure comprised martensite and little-retained austenite. For the CCT diagrams of the specimen with intercritical process, except for the undissolved ferrite phase in each cooling rate zone, the other microstructure types were the same.
(4) Since undissolved ferrites distribute dispersively in an acicular or granular state, they can prevent stress concentration and crack propagation; therefore, the microstructure of the specimen, being quenched at 920 °C and undergoing the intercritical process at 830 °C with the same cooling rate, was much refined, and the strength and toughness were further enhanced.

(5) For the achievement of the bainite + ferrite dual-phase microstructure with well-matched strength and toughness, the cooling rate should be controlled at 1 °C·s\(^{-1}\)–5 °C·s\(^{-1}\).

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