Effect of cooling rate on microstructure and mechanical properties of CB2 tempered martensitic steel

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Abstract: The cooling rate of casting has a significant effect on microstructure and mechanical properties of castings. The 9Cr-1.5Mo-1Co cast steel, referred to as CB2, is one of the most promising alloys for various cast components to be used under ultra-supercritical conditions. In this study, HRTEM, SEM, and XRD methods were used for qualitative and quantitative analyses of contents, phases, and sizes of the inclusions and precipitates, as well as microstructure observation of the tempered martensitic steel at different cooling rates. Traditional tensile tests were conducted to characterize the material mechanical properties. Results show that the size of the boron nitride and precipitate, the width of the martensite lath and the content of the δ -ferrite are reduced as the cooling rate increases from 5–8 °C·min⁻¹ (CB2-S steel) to 50–60 °C·min⁻¹ (CB2-F steel). The precipitates are mainly composed of $M_{23}C_6$ and a small amount of M_3C . The average diameters of the $M_{23}C_6$ precipitates in CB2-F and CB2-S are 202 nm and 209 nm, respectively. The inclusions are mainly composed of BN, Al_2O_3 and MnO_2 , and the inclusion ratios are 0.1969% for the CB2-F and 0.06556% for CB2-S steel. The average martensite lath widths of CB2-F and CB2-S steels are 289 nm and 301 nm, respectively. Furthermore, the M_3C having a diameter of about 150 nm and a thickness of 20 nm is observed in the δ -ferrite of the tempered CB2-S steel. The presence of the δ -ferrite reduces the precipitation strengthening and dislocation density in CB2-S steel. In addition, the lower cooling rate stabilizes the δ -ferrite structure at room temperature.

Key words: tempered martensitic steel; casting; microstructure; $M_{23}C_6$; mechanical properties; inclusions; δ -ferrite

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In recent years, high-chromium heat-resistant ferritic steels with tempered martensite lath structure (TMLS) have been widely used in fossil power plants [1-3]. A new generation of heat-resistant ferritic steels are extensively applied in production of key components of fossil power plants with operating temperatures up to 630 °C [4, 5]. Many researchers have studied the 9Cr-1.5Mo-1Co cast steel, referred to as CB2 [6-8]. CB2 steel containing tempered martensite structure can be used in boiler components being exposed to ultra-supercritical conditions (USC), such as a steam temperature of 630 °C and a pressure of 30 MPa [9-12].

Tempered martensitic heat-resistant steels can be strengthened by solid solution hardening [13], precipitation hardening [14], fine grain hardening [15] and lath hardening [16]. Precipitation hardening and lath hardening are the main strengthening methods for large steel castings to be used under ultra-supercritical conditions. Changes in precipitates and martensite can be achieved through changes in chemical composition and heat treatment. Changes in chemical composition and heat treatment are common approaches to improve steel material properties. The role of precipitates and matrix structure in realization of structural heredity was reported [17, 18]. The effect of cooling rate on microstructure and mechanical properties began to be noticed. It has been reported that the distribution and

size of carbide precipitates are closely related to the cooling rate [19]. Research [20] showed that the morphology and sizes of carbides varied with the increase of the cooling rate of nickel-based superalloys. At low cooling rates, most of the carbides were found to be at the grain boundaries. Rasouli D, et al. [21] indicated that the increase in cooling rate led to the formation of bainite and martensite in the microstructure of microalloyed steel 30MSV6. Jun et al. [22] suggested that during solidification of HSLA steel, the precipitation starts simultaneously during the solidification from liquid to solid, and the precipitation is also dependent on cooling rate. It was found that as the cooling rate increased, the amounts of precipitates gradually decreased, because the diffusion time was insufficient as the cooling rate of the Nb-Ti bearing HSLA steels increased.

During the casting process, the cooling rate determined the structure and properties of the alloy steel [23-24]. At present, the change of solidification rate has become one of the more important means to improve the mechanical properties of materials. However, the influence of cooling rate on the microstructures and mechanical properties of CB2 tempered martensitic steel has not been investigated in detail. In this work, the effects of cooling rate on the microstructure of inclusions, martensite lath, precipitates, and δ -ferrite on the structure of

CB2 steel were studied, and the relationship between cooling rate and mechanical properties of CB2 steel was also discussed.

1 Experimental procedure

CB2 steel samples were prepared at different cooling rates: the laboratory-scale fast cooling (CB2-F) and the industry-scale slow cooling (CB2-S). The CB2-F steel was melted in a medium frequency induction furnace and cast into a 5 kg ingot at 1,650 °C with a cooling rate of about 50–60 °C·min⁻¹. The CB2-S steel supplied by Liaoning Fu'an Heavy Industry Co., Ltd. was melted in an electric arc furnace and then refined by Vacuum Oxygen Decarbonization (VOD). A 500 kg CB2-S steel ingot was prepared by pouring the melt at 1,550 °C into a sand mould and embedding it in the mould for two weeks. The cooling rate of CB2-S steel was about 5–8 °C·min⁻¹. The cooling rates of the experimental steels were calculated by ProCAST software. The ingots of both CB2-F steel and CB2-S steel were cast by top pouring. Both steel samples were austenitized at 1,100 °C for 2 h and then air quenched (AQ). Then, tempering was carried out at 730 °C for 2 h and finally, the samples were subjected to furnace cooling (FC). The chemical compositions of the CB2-F and CB2-S steels are listed in Table 1.

Table 1: Chemical compositions of CB2-F and CB2-S steels (wt.%)

	C	Si	Mn	Cr	Mo	V	Nb	Co	B	N	Ni	O	Fe
CB2-F	0.14	0.21	0.88	9.28	1.41	0.19	0.08	1.03	0.009	0.03	0.2	0.005	Bal.
CB2-S	0.12	0.25	0.92	9.55	1.51	0.19	0.10	1.01	0.011	0.027	0.2	0.003	Bal.

After the heat treatment, the Vickers hardness measurement was conducted with a load of 1 kg and a loading time of 10 s. The reported value is the average of 10–20 measurements. The tensile test specimens were 5 mm gauge diameter and 25 mm gauge length. The tensile tests were carried out at room temperature at an initial strain rate of 0.8 mm·min⁻¹, according to the ISO6892-1: 2009 standard [25].

The carbides, collected by electrolyzing the specimens in 3.6%ZnCl₂ + 5%HCl + 1% citric acid + 90.4% methanol with an electric current density of 0.05 A·cm⁻², were qualitatively analyzed [26] by X-ray diffraction (XRD) using Cu K α radiation. The morphology of martensite and precipitate was observed using an ultra plus scanning electron microscopy (SEM) equipped with an INCA energy dispersive X-ray spectroscopy. The selected area electron diffraction (SAED), crystallinity and morphology of the particles were analyzed by a JEM-2100F high-resolution transmission electron microscope (HRTEM) with a double-tilted specimen holder. HRTEM images were taken from the thin edges of the TEM samples and evaluated by fast fourier transform (FFT) and inverse fast fourier transformation (IFFT). TEM samples were prepared by using double jet electro-polishing with a solution of 8% perchloric acid in glacial acetic acid. Image Pro Plus 6.0 (IPP) was used to calculate the concentration of inclusions in the steel.

2 Results and discussion

2.1 Microstructure

Figures 1 and 2 show the optical microstructure of CB2-F and CB2-S steels before and after heat treatment. It can be seen that the as-cast microstructures of both CB2-F and CB2-S steels consist of inclusions and δ -ferrites. The microstructures after heat treatment consist of tempered martensite and original austenite grain boundaries (PAGB). Multiple martensite blocks are observed inside a grain. Due to the presence of many alloying elements in CB2 steel, the slow cooling rate promotes segregation of alloying elements at the solid-liquid interface. Segregation of alloying elements in CB2 cast steel resulted in high levels of local dislocations. Areas with high levels of local dislocations are preferentially corroded. These corrosion pits are caused by the segregation of the alloying elements shown in Fig. 1(d). After heat treatment, the segregation of the alloying elements disappears, as shown in Fig. 2(b). In general, precipitation of high Cr heat resistant steel occurs in the heat treated matrix, such as PAGB and lath subboundaries. PAGBs can be identified by the different orientations of the lath martensite. Precipitates and lath martensite are the main factors affecting the strength and toughness of alloy steel, and are usually regulated through heat treatment. It has been found that

Fig. 1: As-cast microstructures of CB2-F steel (a, c) and CB2-S steel (b, d)

Fig. 2: Microstructures of heat treated CB2-F (a) and CB2-S (b) steels

the casting cooling rate affects not only inclusions and δ -ferrites, but also precipitates and lath martensite^[21].

2.2 Inclusions

Figure 3 shows the typical morphology of inclusions in CB2-F and CB2-S steels, and the concentration of each element in these inclusions. It is found that the major inclusions in CB2-F steel are boron nitride (BN), alumina (Al_2O_3), MnO_2 and a small amount of (Mn, Si)O, while the major inclusions in CB2-S steel are BN, and a small amount of Al_2O_3 . Most of the inclusions in

CB2-F and CB2-S steels are spherical with average diameters of 6.1 μm and 10.8 μm , respectively. The inclusion volume ratios in CB2-F and CB2-S steels determined by Image Pro Plus 6.0 are 0.20% and 0.066%, respectively. This implies that the content of inclusions in CB2-S steel is much lower than that of CB2-F steel due to the purification capacity of the vacuum oxygen decarburization melting process. In addition, the average diameter of inclusions in CB2-S steel is higher than that of CB2-F steel due to the strong aggregation and growth of inclusions taking place during slow cooling.

Fig. 3: Typical morphology of inclusions in CB2-F (a-c) and CB2-S (d-f) steels and concentrations of elements in inclusions

Fig. 4: Relationship between free energy of BN formation and solidification rate of molten steel

Due to the addition of 0.01% boron and 0.027% nitrogen, coarse BN was formed during solidification process. The solubility of boron in austenite is lower than that in molten steel. The equilibrium partition coefficient (k) of boron in molten steel is approximately 0.001^[27]. During the solidification process, the slow solidification rate causes severe segregation of boron at the solid-liquid interface. As the solidification time increases, the boron content in the molten steel gradually increases (Fig. 4). The free energy of BN formed at the front of the solid-liquid interface in CB2 molten steel is shown in Eq. (1)^[28]. The relationship between the solidification rate f_s and the temperature T_{S-L} of the solid-liquid interface front is established as shown in Eq. (2). The relationship between the solidification rate f_s and the B content $[B]$ of the solid-liquid interface front is established, as shown in Eq. (3). The relationship between the solidification rate f_s and the activity coefficient of the N element f_N of the solid-liquid interface front is established and given in Eq. (4). The relationship between the solidification rate f_s and the activity coefficient of the B element f_B of the solid-liquid interface front

is established as shown in Eq. (5). The relationship between the free energy of BN formation and the solidification rate f_s of steel is presented in Fig. 4. BN reaches the thermodynamic conditions before the molten steel is completely solidified. It has been observed that the faster the casting cools, the greater the number of BNs and the smaller the size of BNs.

$$\Delta G_M = \Delta G^\theta - \Delta \bar{G}^\theta$$

$$= -192,000 + 88.7T_{S-L}$$

$$- 8.314T_{S-L} \ln \left[0.024 \times [B]_x \times f_B \times f_N \right] \quad (1)$$

$$T_{S-L} = 1809 - \frac{38.3}{1 - 0.3919f_s} \quad (2)$$

$$[B]_x = 0.01 \times (1 - f_s)^{0.999} \quad (3)$$

$$f_N = 10^{\left[\left(\frac{3280}{T} - 0.75 \right) \times (0.005876 + 0.094 \times [B]_x) \right]} \quad (4)$$

$$f_B = 10^{\left[\left(\frac{2557}{T} - 0.365 \right) \times (0.040151 + 0.038 \times [B]_x) \right]} \quad (5)$$

2.3 Lath martensite

Figure 5 presents the TEM images of heat treated CB2-F and CB2-S steels. Both CB2-F and CB2-S steels have a tempered martensite lath structure (TMLS). The high strength of martensite steel is mainly due to its high dislocation density. The greater the dislocation density, the greater the strength of the test steel^[29]. The dislocation density of CB2 steel is affected by the width of the martensite lath. The smaller the size of the lath martensite, the greater the dislocation density. Precipitation of carbides in martensite during tempering accompanied

Fig. 5: TEM images of heat treated (a) CB2-F; (b) CB2-S steels

by a decrease in the amount of solid solution elements and in dislocation density. The mixture of precipitates and lath martensites is the structural features of tempered martensite. The lath width of martensite is significantly affected by the cooling rate. For CB2-F and CB2-S steels, the measured average width of the martensite laths is 289 nm and 301 nm, respectively, therefore, the dislocation density of CB2-S steel is lower than that of CB2-F steel.

2.4 Precipitates

As shown in Fig. 6, most of the near-spherical precipitates are distributed at the grain boundaries and only a few in the grains. The size of the precipitates at the grain boundaries is larger than that in the grains. Due to the similar chemical composition, there is no significant difference in the size and concentration of precipitates in CB2-F steel and CB2-S steel. Supersaturated C and Cr elements precipitated in the grains and at the lath

boundaries while forming Cr-rich carbides. Carbides precipitated at the lath boundaries can reduce the strength of martensite. The X-ray diffraction measurements (Fig. 7) show that the carbides are $M_{23}C_6$ in both CB2-F and CB2-S steels. $M_{23}C_6$ is the main precipitate of tempered high chromium heat-resistant steels, where M corresponds to Cr element. After tempering at 730 °C, $Cr_{23}C_6$ forms in both CB2 steels. This indicates the cooling rate does not change the type of precipitates in the CB2 steels.

Figure 8 presents the size distribution of $M_{23}C_6$ in CB2-F and CB2-S steels. The particle size distribution of precipitates in CB2-F steel is more concentrated. In CB2-F and CB2-S steels, the average diameters of the $M_{23}C_6$ carbides are 202 nm and 209 nm, respectively. It implies that the size and amount of carbides in the steels are independent of inclusions. Size and amount of carbides in the steels are dependent of density of the grain boundary. The density of the grain boundary can

Fig. 6: SEM images of heat treated CB2-F (a-b) and CB2-S (c-d) steel samples

Fig. 7: X-ray diffraction patterns of heat treated CB2-F and CB2-S steel samples

Fig. 8: Size distribution of $M_{23}C_6$ carbides in CB2-F and CB2-S steel samples

be increased by refinement of the prior austenite grains, which provides additional space for precipitation of carbides.

Figure 9 shows TEM images of carbides in heat treated CB2-S steel. In most cases, carbides ($M_{23}C_6$) are located in the grains and at the lath boundaries. The FFT implies that the obtained $M_{23}C_6$ has a face-centered cubic structure, and the EDS results demonstrate that $M_{23}C_6$ consists of Fe, C, Cr and Mo. Due to the low nucleation interfacial energy of the original austenite boundaries and martensite laths^[30], $M_{23}C_6$ carbides were transformed from M_3C and then precipitated. Dislocation motion was blocked by fine carbides. Fine carbides play an important role in controlling the subgrain coarsening during high temperature creep. The stability of the subgrain boundaries is mainly due to the fact that the smaller-sized $M_{23}C_6$ is close to the boundaries and provides Zener forces^[31].

2.5 δ -ferrite

In CB2-F steel (Fig. 1c), some inclusions of about 2 μm are observed, while no δ -ferrite is observed. In CB2-S steel, a δ -ferrite phase of about 20 μm is observed (Fig. 1d), while no inclusions are observed. CB2 steel is a high alloy steel. As the solidification rate of molten steel decreases, the segregation degree of alloying elements gradually increases. The segregation of alloying elements provides thermodynamic conditions for the δ -ferrite residues. The δ -ferrite content in CB2-S steel is less than 4%, as shown in the optical micrographs of Fig. 1 (d). The presence of δ -ferrite reduces the creep resistance of the heat-resistant steel^[32]. It is generally noted that many microalloying elements (Nb and V) do not improve the creep resistance of heat-resistant steel due to the presence of δ -ferrite^[32]. Therefore,

Fig. 9: (a) TEM image of $M_{23}C_6$ in CB2-S steel sample; (b) HRTEM image of $M_{23}C_6$; (c) EDS spectrum of $M_{23}C_6$ particle; (d) FFT diffraction spots, which are used to reconstruct the HRTEM image given in (b)

as the cooling rate increases, the δ -ferrite content decreases and the creep strength increases. On the other hand, the slow cooling rate increases the degree of segregation of alloying elements in high alloy steels, which results in more δ -ferrite residues.

It is well known that four types of carbide precipitations form during tempering of chromium steels: ϵ -carbide, cementite (M_3C), M_7C_3 , and $M_{23}C_6$ ^[30]. Typically, M_3C precipitates in the carbon steel at a lower tempering temperature or during an early tempering stage^[33]. However, it is found that after tempering at 730 °C, M_3C (M corresponds to Fe or Cr atoms) is formed

in the δ -ferrite in CB2-S steel (Fig. 10). M_3C is only present in δ -ferrite having a low total content. When the tilt angle of the sample stage is changed, the morphology of M_3C changes from a needle-like shape to a flake-like shape. The diameter and thickness of M_3C are about 150 nm and 20 nm, respectively, as shown in Fig. 11. $M_{23}C_6$ precipitates at the boundary of martensite lath and M_3C precipitates in δ -ferrite. This indicates that the contents of Cr and Mo in δ -ferrite are extremely low, and the condition for forming $M_{23}C_6$ is not satisfied in δ -ferrite during the tempering process.

Fig. 10: (a) IFFT micrograph of M_3C in δ -ferrite; (b) FFT diffraction spots, which are used to reconstruct IFFT image given in (a)

Fig. 11: (a) SEM micrograph of δ -ferrite in CB2-S steel; (b) TEM micrograph of δ -ferrite in CB2-S steel and (c-d) M_3C in δ -ferrite with different tilt angles

2.6 Mechanical properties

Tensile test and Vickers hardness measurement of CB2-F and CB2-S steels were conducted at room temperature, and the results are shown in Fig. 12. The tensile strengths of CB2-F and CB2-S are 793 MPa and 757 MPa, and the hardness values are 252 HB and 248 HB, respectively. The strength of CB2-S steel is lower than that of CB2-F steel, which is mainly affected

by the large size of precipitates, the wide lath martensite and δ -ferrite content. The elongations of CB2-F and CB2-S are basically the same at 15.85% and 15.23%, respectively. This means that the cooling rate has an effect on the tensile strength of CB2 steel and has virtually no effect on elongation and hardness. The tensile stress-strain curves of CB2-F and CB2-S steels are shown in Fig. 13.

Fig. 12: Tensile strength, yield strength and hardness of CB2-F and CB2-S steels

Fig. 13: Tensile stress-strain curves of CB2-F and CB2-S steels

It is well known that the basic methods for strengthening martensitic heat-resistant steels are solid solution strengthening, precipitation hardening, fine grain hardening and lath hardening. Based on previous research^[34], the solid solution strengthening effect is small as compared to precipitation hardening and lath hardening. In order to further clarify the effect of cooling rate on precipitation hardening and lath substructure hardening, the following equations are used^[34]:

$$\sigma_p (\text{MPa}) = \frac{5.9\sqrt{f}}{\bar{x}} \times \ln \left(\frac{\bar{x}}{2.5 \times 10^{-4}} \right) \quad (6)$$

$$\sigma_l (\text{MPa}) = \frac{10Gb}{\lambda_1} \quad (7)$$

where f refers to the volume fraction of the precipitates, $\bar{x}$ corresponds to the average diameter of the particles in microns, G refers to the shear modulus, b represents the length of the Burgers vector and λ_1 corresponds to the average lath width. The shear modulus and the Burgers vector in Eq. (2) are 81,600 MPa and 0.248 nm, respectively^[35]. The strengths of precipitates (σ_p) and laths (σ_l) in CB2-F and CB2-S steels are listed in Table 2. The influence of the cooling rate on the mechanical properties of CB2 steels is consistent with the results given in Table 2.

Table 2: Strengths of precipitates (σ_p) and laths (σ_l) in CB2-F and CB2-S steels

	CB2-F	CB2-S
σ_l (MPa)	700.48	671.29
σ_p (MPa)	52.01	43.83

3 Conclusions

The effect of cooling rate on the microstructure and mechanical properties of CB2 steel was investigated. The major findings of this work are summarized below:

(1) The inclusions are mainly composed of BN, Al_2O_3 , MnO_2 and a small amount of (Mn, Si)O in CB2-F (the laboratory-scale fast cooling) 9Cr-1.5Mo-1Co cast steel, and BN and Al_2O_3 in CB2-S (the industry-scale slow cooling) 9Cr-1.5Mo-1Co cast steel. The concentration of inclusions in CB2-S steel is lower than that of CB2-F steel, whereas the size of inclusions in CB2-S steel is larger than CB2-F steel.

(2) After tempering at 730 °C, δ -ferrite is observed in CB2-S steel, and M_3C particles having a diameter of about 150 nm and a thickness of about 20 nm are formed in δ -ferrite, which reduced the effect of precipitation strengthening on CB2-S steel.

(3) The faster the CB2 steel cools, the better the precipitation strengthening and the lath hardening effect on it. The strength of laboratory-scale CB2-F steel is greater than that of industry-scale CB2-S steel. The elongations of CB2-F and CB2-S are basically the same.

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