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Effect of tempering temperature on microstructure and mechanical properties of high boron white cast iron

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Abstract: The effect of different tempering temperatures on the microstructure and mechanical properties of airquenched high boron white cast iron was studied. The results indicate that the high boron white cast iron comprises dendritic matrix and inter-dendritic M_2B boride; and the matrix comprises martensite and pearlite. After quenching in the air, the matrix is changed into lath martensite; but only 1-µm-size second phase exists in the matrix. After tempering, another second phase of several tens of nanometers is found in the matrix, and the size and quantity increase with an increase in tempering temperature. The two kinds of second precipitation phase with different sizes in the matrix have the same chemical formula, but their forming stages are different. The precipitation phase with larger size forms during the austenitizing process, while the precipitation phase with smaller size forms during the tempering process. When tempered at different temperatures after quenching, the hardness decreases with an increase in the tempering temperature, but it increases a little at 450 °C due to the precipitation strengthening effect of the second phase, and it decreases greatly due to the martensite decomposition above 450 °C. The impact toughness increases a little when tempered below 300 °C, but it then decreases continuously owing to the increase in size and quantity of the secondary precipitate above 300 °C. Considered comprehensively, the optimum tempering temperature is suggested at 300 °C to obtain a good combination of hardness and toughness.

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mong iron-based wear-resistant materials, there have been a lot of research and applications for white cast iron that takes carbide as wear-resistant phase; however, low toughness limits their application range. In recent years, much attention has been paid to iron-based alloys that include boride in the wear-resistant phases. Compared with carbide, boride is much harder and has better heat stability ^[1], so it can be used as a wear-resistant phase. From the 1980s, there appeared some research papers and patents about this kind of material. Aso et al. ^[2,3] studied the solidification process, phase diagram, and phase transformation of Fe-Cr-C-B alloys. Lakeland [4,5] invented a kind of alloy that uses boride as wear-resistant phase and can be used to make glass molds and rollers. Egorov et al.^[6] studied the effect of different carbon contents on the hardness and heat stability of iron-based alloys that include boride in the wear-resistant phases. Gol'dshtein et al.^[7] studied

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some properties of alloys that include boride in the wearresistant phases. In China, some research has been performed by Liu^[8], Fu^[9, 10] and Guo^[11-14]. The microstructure and mechanical properties of such kinds of alloy were analyzed, and some products, such as wear-resistant linings, rollers and other wear-resistant parts, have resulted from this research^[12-14].

High boron white cast iron is a new kind of wear-resistant white cast iron that includes boride in the wear-resistant phase. The advantages of this kind of white cast iron are that the matrix and the quantity of boride can be controlled by boron and carbon, as carbon can not be dissolved in boride. Therefore, it is possible to get high toughness of matrix with low carbon content. The matrix is the resource of toughness; regarding white cast iron with carbide, it is difficult to get a low carbon matrix, so even if the distribution of carbide can be separated, the toughness is still low, as the matrix is always high carbon martensite. High boron white cast iron characterizes low carbon and high boron in terms of composition, which has a better combination of hardness and toughness than traditional white cast iron with carbide. So far, high boron white cast iron has been used to make some products, and good results have been achieved.

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In this paper, the effects of different tempering temperatures on the microstructure and mechanical properties were studied, and an optimum tempering temperature was suggested for application.

1 Experimental procedure

The chemical composition of the alloy is presented in Table 1.

Table1: The chemical composition of alloy (wt.%

В	С	Si	Mn	Cr	Cu	Ti	AI	Fe
1.62	0.32	0.46	0.61	10.85	0.21	0.024	0.039	Bal.

High boron white cast iron was melted in a 100-kg medium frequency coreless induction furnace with SiO₂ lining, with charge materials of steel scrap, graphite, Fe-B, Fe-Mn, Fe-Cr, Fe-Si master alloys and Cu. As boron is an active element, oxygen and nitrogen in the melt should be removed effectively to ensure the yield of boron. So Al wire and Fe-Ti were added to remove oxygen and fix nitrogen before the Fe-B alloy was added in. The melt was superheated to 1,550 °C, and then poured into Y-blocks made by investment casting.

All samples were cut from the lower part of the Y-blocks. The heat treatment of the samples was carried out in an electrical resistance furnace. Samples were held at 1,020 $^{\circ}$ C for 2 h, quenched in the air, and then tempered at 200, 300, 400, 450, 550, 600, and 650 $^{\circ}$ C, respectively, for 1 h. After heat treatment, the samples were machined to 20 mm × 20 mm × 110 mm.

Impact tests were done using an 150-J capacity machine at room temperature. The impact toughness values reported in this paper are averages of three tests. Hardness was tested on a Rockwell hardness machine. Five readings were taken on each sample and the average of them is reported here. Dilatometer test was carried out using a Gleebe-800 testing machine, and the size of the sample is ϕ 8 mm × 120 mm. The microstructures of the samples were examined using a Neophot 32 optical microscope (OM) and an FEI Quanta 200 FEG scanning electronic microscope (SEM) with energy dispersive analysis by X-ray (EDAX) after the samples were etched with 10vol.%HNO₃ + 3vol.%HCl + 10vol.%FeCl₃ (saturated) + 77vol.%ethanol. The X-ray diffraction (XRD) analysis was performed on a D/max-RB X-ray diffractometer to determine the boride type. The specimens were scanned using Cu Ka radiation at 40 kV and 300 mA. The scanning speed (2θ) was 1°·min⁻¹.

2 Results and discussion

2.1 As-cast microstructure

Figure 1 shows the as-cast microstructure of high boron cast iron. In its as-cast condition, high boron white cast iron comprises a dendritic matrix and inter-dendritic eutectic. The eutectic compound is M_2B according to the XRD pattern, as shown in Fig. 2, and M represents Fe, Cr or Mn (see Fig. 3). The matrix comprises two different microstructures, i.e., pearlite and martensite (see Fig. 1).



(a) OM micrography

(b) SEM micrography





Fig. 2: X-ray diffraction pattern of as-cast high boron white cast iron



Fig. 3: EDAX spectra of boride in high boron white cast iron

2.2 Microstructure after heat treatment

According to the dilatometer curve (Fig. 4), the phase transformation of high boron white cast iron begins at about 850 $^{\circ}$ C and ends at about 900 $^{\circ}$ C; and the martensite forms at about 347 °C and ends at 245 °C. Therefore, we decided on 1,020 °C as the austenitizing temperature in the experiment. After quenching, the samples were tempered at 200, 300, 400, 450, 500, 550, 600 and 650 °C for 1 h. Figure 5 shows the



Fig. 4: Dilatometer curve for high boron white cast iron starting from as-cast state

microstructure after quenching without tempering. Figures 6 and 7 show the microstructure after quenching with tempering at 200 °C and 650 °C, respectively.

After quenching, the morphology of boride remains almost unchanged, but the matrix changes into lath martensite. In addition, some small second phase appears in the matrix, with a size of about 1 µm (Fig. 5). In the case of the tempered condition, another much smaller second phase appears, and the size is below 100 nanometers, the amount of which increases as the tempering temperature is increased. The XRD pattern (Fig. 8) shows that the two kinds of particles have the same chemical formula of M₂₃(C, B)₆, where M may represent Fe, Cr or Mn. Compared with other alloy steel, the tempering carbide is $M_{23}(C, B)_6$ other than $M_3(C, B)$, which can be explained in terms of boron content and free energy. On the one hand, the boron content in the matrix is higher than that in the common boron steel; on the other hand, the free energy of $M_{23}(C, B)_6$ is lower than that of $M_3(C, B)^{[15]}$, so the former is easy to precipitate. Regarding to as-cast condition, eutectoid reaction happens to form pearlite when cooled from the liquid condition, so the carbide is $M_3(C, B)$ type.

Comparing Fig. 5 with Figs. 6 and 7, the two different sizes



(b) High magnification





(a) Low magnification

(b) High magnification

Fig. 6: Microstructure of high boron white cast iron held at 1,020 °C for 2 h, quenched in air, tempered at 200 °C for 1 h



Fig. 7: Microstructure of high boron white cast iron held at 1,020 °C for 2 h, quenched in air, tempered at 650 °C for 1 h



of the second phase are formed by different processes. The larger size particles are formed in the process of austenitizing, and the smaller ones are formed in the process of tempering. The two different sizes of the second phase precipitation are due to boron diffusion, and the larger one forms at high temperature, whereas the smaller one forms at low temperature, which results in the size difference.

2.3 Effect of different tempering temperatures on mechanical properties

Figures 9 and 10 show the hardness and impact toughness, respectively, at different tempering temperatures after quenching.

As the tempering temperature increases, the hardness decreases at first, and then increases a little at 450 °C, but then decreases a lot when the temperature is higher than 450 °C. When the tempering temperature is below 450 °C the hardness decreases little by little due to the decomposition of martensite. When at 450 °C, a large amount of the smaller second phase appears, and results in the higher hardness due to the precipitation strengthening effect. Over 450 °C, the martensite is mostly decomposed, which results in the serious decrease in hardness.

For impact toughness, the variation is different. At the



temperatures after quenching



Fig. 10: Impact toughness at different tempering temperatures after quenching

beginning, the impact toughness increases as the tempering temperature increases, reaches a maximum value at 300 $^{\circ}$ C, and then decreases continuously for temperatures over 300 $^{\circ}$ C. Below 300 $^{\circ}$ C, the decomposition of martensite leads to the impact toughness increasing. However, for temperatures over 300 $^{\circ}$ C, the impact toughness seriously decreases owing to the precipitation and aggregation of the second phase. According to the experimental results, high boron white cast iron has the best combination of hardness and toughness at a tempering temperature of 300 $^{\circ}$ C.

3 Conclusions

(1) The as-cast microstructure of high boron white cast iron is composed of a dendrite matrix and inter-dendrite eutectic boride M_2B . The matrix is composed of martensite and pearlite.

(2) After quenching in the air, the matrix changes into martensite. Without tempering, there only exits a second precipitation phase with the size of about 1 μ m. When tempered, a smaller second phase below 100 nanometers appears, and the quantity increases as the tempering temperature increases. The two different sizes of second phase are formed by different processes. The larger one forms in the process of austenitizing, and the smaller one forms in the process of tempering.

(3) When tempered at different temperatures after quenching, the hardness increases a little at 450 $^{\circ}$ C due to second phase strengthening; but all other tempering temperatures lead to the hardness decreasing. At 300 $^{\circ}$ C the impact toughness reaches a maximum value. Through comprehensive analysis high boron white cast iron has the best combination of hardness and toughness when tempered at 300 $^{\circ}$ C.

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