A Study on the Microstructures and Toughness of Fe-B Cast Alloy Containing Rare Earth

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This study investigates the effect of cerium on the microstructures, mechanical properties of medium carbon Fe-B cast alloy. The as-cast microstructure of Fe-B cast alloy consists of the eutectic boride, pearlite, and ferrite. Compared with the coarse eutectic borides in the unmodified alloy, the eutectic boride structures in the modified alloy are greatly refined. Cerium promotes the formation of Ce_2O_3 phase. Ce_2O_3 can act as effective heterogeneous nuclei of primary austenite, and refine austenite and boride. After heat treatment, the impact toughness of the modified alloy is higher than that of the unmodified alloy because there are more broken borides in the modified alloy. Meanwhile, the fracture mechanism of medium carbon Fe-B alloy is depicted and analyzed by using fractography.

Keywords boride, cerium, impact toughness, microstructure

1. Introduction

High chromium white cast irons have superior wear resistance and are widely used in mineral processing, cement production, thermal electric power plants, and others (Ref 1-3). Their exceptional wear resistance is the result of their high carbon and chromium concentrations, which form $(Cr,Fe)_7C_3$ carbides along with austenite during solidification. However, high chromium white cast irons suffer from shortcomings such as high concentration of alloying elements, exorbitant production cost, and propensity for deformation and fracture after heat treatment (Ref 4). So, the development of new type wear-resistant material is very important.

The studies of boronizing reveal that a Fe₂B layer produces superior wear resistance and mechanical properties (Ref 5, 6). This makes it possible to apply Fe₂B as the wear-resistant phase. According to Fe-B phase diagram (Ref 7), boron has a very low solubility in α -iron (0.0081 wt.%) and γ -iron (0.02 wt.%). So, this makes the formation of boride possible when boron is added in iron melt. Because of high boron concentration, the solidification process of Fe-B alloy is summarized as follows: First, the primary γ phase separates out from the melt. Since the partition coefficients of elements

Dawei Yi and **Zhiyun Zhang**, School of Materials Science and Engineering, Xi'an University of Science and Technology, 58 Yanta Road, Xi'an 710054 Shaanxi Province, People's Republic of China; **Hanguang Fu**, Research Institute of Advance Materials Processing Technology, School of Materials Science and Engineering, Beijing University of Technology, Beijing 100124, People's Republic of China; **Chengyan Yang**, Foundry, Zibo Diesel Engine Company, Zibo 255077 Shandong Province, People's Republic of China; and **Shengqiang Ma** and **Yefei Li**, State Key Laboratory for Mechanical Behavior of Materials, School of Materials Science and Engineering, Xi'an Jiaotong University, Xi'an 710049 Shaanxi Province, People's Republic of China. Contact e-mail: yidawei19820608@163.com. such as chromium, manganese, and boron are lower than one in γ phase (Ref 8), these elements will be discharged into melt accompanied by the growth of the γ phase. When the boron concentration in the melt nears 3.8 wt.%, the eutectic reaction occurs in the remnant melt (Ref 7). Therefore, the addition of boron promotes the formation of eutectic boride.

Moreover, the boride has high hardness, and therefore, can be taken as the strengthening phase. This information would be helpful to develop a new type of wear-resistant material. The conspicuous advantage of Fe-B alloy is that the matrix and boride could be controlled, respectively, by carbon and boron contents. In this case, the strengthening and toughening matrix and adequate volume percentage of boride could be obtained at the same time, which would make it possible to design the microstructure that we need (Ref 9).

Fe-B alloy has such characteristics as excellent wear resistance, low alloying content, simple casting process, and low production costs (Ref 10, 11). The application of the Fe-B alloy is helpful for the decrease of material consumption due to its high abrasion resistance and toughness (Ref 12). However, there exist many coarse borides, which are not favorable to the improvement of the strength and toughness of Fe-B alloy. Fu et al. (Ref 13) studied the effects of rare earth and titanium additions on the microstructures and properties of low carbon Fe-B alloys, and pointed out that proper modification using rare earth combined with titanium can refine the grain size of primary austenite and increase the impact toughness of the Fe-B alloy. However, the refinement mechanism of rare earth in the Fe-B alloy should be further studied.

The purpose of present research is to study the microstructures and impact toughness of a Fe-B cast alloy modified by cerium. As a result, the as-cast eutectic boride microstructures are greatly refined, and the impact toughness increases when cerium is added.

2. Experimental Procedures

2.1 Melting, Casting, and Heat Treatment of Fe-B Cast Alloy

The alloys used for this investigation were melted in a 10 kg capacity medium-frequency induction furnace using a quartzite

lining. Initial charge materials contained clean pig iron and steel scrap. When these initial charge materials were melted, Fe-61.5 wt.% Cr alloy, Fe-65.8 wt.% Mn alloy, Fe-75.3 wt.% Si alloy, and copper were added to molten alloy. When the temperature of molten alloy was up to 1610 °C, Fe-16.1 wt.% B alloy, and Fe-26.5 wt.% Ti alloy were added to the melt. After deoxidation by pure aluminum, the molten alloy was modified using Fe-63.4 wt.% Cr-12.6 wt.% N alloy and Fe-39.4 wt.% Ce-24.2 wt.% Si alloy when it was poured into the ladle (before pouring, the modifier had been placed in the ladle) and then was poured at 1470 °C into a sand mold. The specimens were austenized at 1050 °C for 4 h using box-type resistance furnace (with argon protection), followed by quenching in water. Subsequently, the specimens were tempered at 180 °C for 4 h. The chemical compositions of the alloy, determined by optical emission spectroscopy, are shown in Table 1.

2.2 Microstructure Examination

The geometry of sand mold and specimens is shown in Fig. 1. The red rectangle (in Fig. 1b) of approximate dimension

 $20 \times 20 \times 10$ mm was sliced. The metallographic analysis was carried out on the "polished surface" (the polished surface is 40 mm to the bottom of rectangle sample, see Fig. 1b). Before this analysis, the polished surface was etched by 5% nital. Furthermore, the microindentation hardness was also measured at this polished surface.

The metallographic analysis techniques used for material structure included optical microscope (OM), scanning electron microscope (SEM), x-ray diffraction (XRD), and transmission electron microscope (TEM). The OM employed was Neophot 21. The SEM employed was a JEOL JSM-6360LV and a Noran System Six microscope equipped with an energy dispersive x-ray spectrometry (EDS). XRD was performed on a D/MAX-2400 diffractometer with Cu K α radiation at 40 kV and 100 mA as an x-ray source. The specimen was scanned in the 2 θ range of 20°-85° in a step scan mode (0.02° per step). The TEM used was a JEM-200CX, and the TEM specimens (the diameter is 3 mm and the thickness is 50 µm) were prepared by electrolytic polishing at 30 V, using a solution of 5% perchloric acid and 95% methanol at 0 °C.

Table 1 Chemical compositions of Fe-B alloys (wt.%)

Specimens	С	В	Cr	Cu	Mn	Si	Ce	Fe
Co	0.31	1.05	1.10	0.46	0.81	0.51	0	Bal.
C ₁	0.27	1.08	1.06	0.42	0.79	0.58	0.13	
$\dot{C_2}$	0.31	1.08	1.11	0.52	0.84	0.55	0.36	
C ₃	0.28	1.07	1.07	0.47	0.86	0.61	0.54	



Fig. 1 Schematic (unit in mm) of sand mold (a) and specimens (b) (Color figure online)



Fig. 2 Solidification microstructures of as-cast Fe-B alloys: (a)-(d) showing 200 times magnification structures of C_0 , C_1 , C_2 , and C_3 alloys, respectively; 1—ferrite; 2—pearlite; 3—Fe₂B

Leica digital image analyzer was used for analyzing the number of grains per square millimeter (n_a) . The number of grains per square millimeter (n_a) in one image, measured by area method (a circle of 50 mm diameter), is gained from (Ref 14):

$$N = N_{\text{Inside}} + \frac{1}{2}N_{\text{Intercepted}} - 1 \tag{Eq 1}$$

$$n_{\rm a} = \frac{M^2 \cdot N}{A_0},\tag{Eq 2}$$

where N_{Inside} is the number of grains completely inside the test circle, $N_{\text{Intercepted}}$ is the number of grains that intercept the test circle, N is the number of grains related to the test circle, M is the magnification of images, and A_0 is the area of the test circle.

At a magnification of 50 times, 20 images of the specimen were randomly selected to calculate n_a , and the test result was the average value of 20 measurements.

2.3 Hardness and Impact Toughness Tests

Microindentation hardness was measured on a HXD-1000TMC tester according to American Society for Testing and Materials (ASTM) standard E384-08 (Ref 15), and 0.98 and 0.49 N were the applied loads for borides and matrix, respectively. The full test force was kept for 10 s. Twenty indentations were made on each specimen to check the reproducibility of hardness data. The HRS-150 Rockwell hardness tester was used to measure the room temperature hardness, and the applied load was 1470 N. The test result was the average value of ten measurements.

The unnotched impact tests were performed at room temperature by JB-300B impact testing device. The anvil span length was 70 mm, and the impact energy was 300 J. The unnotched impact specimen size (rectangle sample) was $20 \times 20 \times 110$ mm. The impact toughness value was the average of six specimens.

3. Results and Discussion

3.1 Solidification Microstructures of Fe-B Alloy

The microstructures of Fe-B alloys with different cerium concentrations are exhibited in Fig. 2. Figure 3 shows the XRD spectrum of C_0 , C_1 , C_2 , and C_3 alloys. Figure 4 shows the microindentation hardness of metallic matrix in Fe-B alloys. Figure 2 indicates that the microstructures of Fe-B alloys are composed of the eutectic boride and metallic matrix (Ref 16). The eutectic boride is proved to be Fe₂B (area "3" in Fig. 2) by XRD (Fig. 3a-d); while the metallic matrix is composed of the ferrite (area "1" in Fig. 2) and pearlite (area "2" in Fig. 2) because the hardness values are in accordance with their natural hardnesses (Ref 16).

The backscattered electron (BSE) micrographs of C_1 and C_3 alloys are shown in Fig. 5. Figure 5 displays that the number of



Fig. 3 X-ray diffraction spectrum of as-cast alloys

cerium compounds (white particles) increase with the increase of cerium concentration (Fig. 5a and b). The corresponding EDS spectrum at point A in Fig. 5(c) reveals that Ce and O elements are present. Figure 6 shows the bright field TEM micrograph and corresponding selected area diffraction pattern of cerium compound in C_3 alloy. The electron diffraction pattern proves that the dark region is close-packed hexagonal

structure. These results indicate the existence of Ce_2O_3 particles in Fe-B alloy modified by cerium (Ref 17).

The number of grains per square millimeter (n_a) of Fe-B alloys is shown in Fig. 7. It can be seen from Fig. 7 that the alloys containing cerium have higher n_a than that in the unmodified alloy (C₀ alloy), and n_a of C₂ alloy is slightly lower than that of C₃ alloy. The refinement of the eutectic boride



Fig. 4 Microindentation hardness of metallic matrix in Fe-B alloys



Fig. 6 Bright field transmission electron microscope micrograph and corresponding selected area diffraction pattern of cerium oxide in as-cast C₃ alloy



Fig. 5 SEM and distribution of cerium in Fe-B alloys: (a) showing BSE micrograph of C_1 alloy; (b) and (c) showing BSE micrographs of C_3 alloy; (d) showing EDS spectra of point A (c)

would be promoted by the fine matrix grains. So, the microstructures of C_0 alloy (Fig. 2a) show the coarse eutectic borides along grain boundaries. Compared with C_0 alloy, more refined eutectic borides in modified alloys are obtained (Fig. 2b-d). Some researchers studied the microstructure of

Fe-B cast alloy modified by neodymium (Ref 18), their result showed that the neodymium could refine the as-cast microstructure. These results reveal that the rare earth (neodymium and cerium) in Fe-B alloy should be effective modifiers for microstructure refinement.



Fig. 7 The number of grains per square millimeter of Fe-B alloys



Fig. 9 Microindentation hardness of boride in Fe-B alloys



Fig. 8 Heat-treated microstructures of Fe-B alloys: (a)-(d) showing 500 times magnification microstructures of C₀, C₁, C₂, and C₃ alloy, respectively; 1—martensite; 2—Fe₂B (Color figure online)



Fig. 10 Schematic diagram of boride network in Fe-B alloy



Fig. 11 Rockwell hardness and impact toughness of Fe-B alloys

3.2 Heat-Treated Microstructures of Fe-B Alloy

The microstructures of heat-treated C_0 , C_1 , C_2 , and C_3 alloys are shown in Fig. 8. Figure 9 displays the microindentation hardness of the boride. The microindentation hardness analysis (Fig. 4 and 9) reveals that the eutectic boride and metallic matrix would be Fe₂B (white phase in Fig. 8) and martensite (black phase in Fig. 8) (Ref 19). Moreover, compared with as-cast metallic matrix (ferrite and pearlite), the heat-treated metallic matrix (martensite) has higher microindentation hardness.

It can be seen from Fig. 8(a) that a few of eutectic boride networks are broken in C_0 alloy. Compared with C_0 and C_1 alloy, there are more broken borides in C_2 and C_3 alloy, and their morphology is short rod-shaped and round (the red arrows in Fig. 8c and d). Generally, the number of broken borides increases with the increase of n_a (the number of grains per square millimeter). These results prove that cerium is helpful for breakdown of the eutectic boride during heat treatment.

During heat treatment, the boride in Fe-B alloy changes from strip morphology to spheroidized morphology. The thermodynamic driving force comes from the decrease of free energy between eutectic boride and matrix interface. The kinetic driving force is the diffusion of the boron atom on the boride/matrix interface. At high temperature, the diffusion of boron atoms along the boride/matrix interface results in part of the eutectic boride dissolving in the matrix and then precipi-



Fig. 12 Fractographies of impact test specimens: (a) C_0 specimen; (b) C_2 specimen (Color figure online)

tating on boride itself (Ref 20). So, the spheroidized boride forms during high temperature heat treatment. The schematic diagram of the boride network in Fe-B alloy is shown in Fig. 10. It can be seen that the plane coarse joint and thin weak joint exist in the boride network. The diffusion of boron at the thin weak joint (low W_2) is easier and faster than at the plane coarse joint (high W_1) (Ref 20, 21), therefore, the breaking of the fine boride network in modified alloy is easier than coarse boride network in unmodified alloy (C₀).

3.3 The Hardness and Impact Toughness of Fe-B Alloy

The Rockwell hardness and impact toughness of Fe-B alloys with different cerium concentrations are shown in Fig. 11. From Fig. 11, it can be seen that the Rockwell hardness increases slightly with rising cerium concentration. The increasing hardness could be caused by the microstructure refinement. Moreover, the impact toughness value increases with the increase in cerium concentration. Figure 8 and 11 reveal that the more the number of broken borides, the higher is the impact toughness value.

The fractographies of C_0 and C_2 impact specimens are shown in Fig. 12. The theory of the fracture mechanics points out that, the brittle fracture includes initiation and propagation of the crack (Ref 22), which is determined by the matrix and the amount, size, and morphology of the borides in the Fe-B alloys. Because all the metallic matrices of heat-treated Fe-B alloys are martensitic, the initiation and propagation of the crack will be determined by the amount, size, and morphology of the borides.

In C_0 alloy, the boride networks are coarse and concentrated (Fig. 8a), the cracks are easy to form and propagate along the coarse borides, and some big cleavage planes are left on the fracture (see the red circle in Fig. 12a). In this case, the impact toughness will be low. However, in C_2 alloys, the breaking of boride networks is apparent, and the broken borides are fine and dispersed (Fig. 8c). The appearance of local broken boride networks effectively. In this situation, the big cleavage plane will not appear (Fig. 12b), and high impact toughness value is realized (Ref 19).

3.4 The Refinement Mechanism of Cerium on the Boride

Rare earth elements can act to deoxidise the steel, and decrease the content of oxygen by combining with them to form rare earth compounds (Ref 17). In the present study, from Fig. 5 and 6 (SEM, EDS, and TEM), the existence of Ce₂O₃ phase in Fe-B alloys (with cerium) has been proved. According to the formula in Ref 23, the ΔG of Ce₂O₃ at the temperature of 1500 °C is -688,606 J/mol, which indicates that Ce₂O₃ can be formed at the temperature of 1500 °C and can exist stably in the melt when cerium is added. This calculation reveals that Ce₂O₃ particles can precipitate before the formation of primary austenite (Ref 13, 23).

The value of the two-dimensional lattice misfit is used to estimate whether Ce_2O_3 can act as the heterogeneous nuclei of primary austenite. A mathematical model of the two-dimensional lattice misfit (Ref 24) is

$$\delta_{(hkl)_{s}}^{(hkl)_{s}} = \sum_{i=1}^{3} \frac{|d_{[uvw]_{s}}^{i}\cos\theta - d_{[uvw]_{s}}^{i}|/d_{[uvw]_{s}}^{i}}{3} \times 100,$$
 (Eq 3)

where the $(hkl)_s$ is the low index crystal face of the nucleus, the $[uvw]_s$ is the low index crystal orientation in the $(hkl)_s$, the $(hkl)_n$ is the low index crystal face of the new crystal phase, the $[uvw]_n$ is the low index crystal orientation in the $(hkl)_n$, $d_{[uvw]_s}$ and $d_{[uvw]_n}$ are the atomic spacing along the $[uvw]_s$ and the $[uvw]_n$, respectively, θ is the angle included between the $[uvw]_s$, and the $[uvw]_n$ ($\theta < 90^\circ$). Bramfitt proposed a theory (Ref 25) that during heterogeneous nucleation, the nuclei with $\delta < 6\%$ are the most effective, while those with δ between 6 and 12% are medium effective and those with $\delta > 12\%$ are ineffective.

The structure of Ce₂O₃ is close-packed hexagonal. Its lattice parameters are as follows: a = 0.3927 nm and c = 0.6062 nm. The primary austenite (γ) has a face-centered cubic structure. The lattice parameter of primary austenite is 0.3726 nm. The calculation reveals that the lattice misfit between $(0001)_{Ce,O_3}$ and $(111)_{\gamma}$ is 5.4% (Ref 26). Consequently, Ce₂O₃ can act as effective heterogeneous nuclei of primary austenite and facilitate the refinement of austenite. The fine austenite will promote the refinement of borides during solidification, and the fine borides are in favor of broken boride network appearance during heat treatment (Ref 13).

4. Conclusions

The eutectic borides in medium carbon Fe-B cast alloy were refined by cerium addition. This work demonstrates the following conclusions:

- (1) The as-cast microstructures of Fe-B alloys are composed of ferrite, pearlite, and eutectic boride. The number of grains per square millimeter in Fe-B alloys with cerium addition is higher than that in unmodified alloy. Moreover, compared to the coarse eutectic borides in unmodified alloy, the eutectic boride in Fe-B alloys modified by cerium are greatly refined.
- (2) After heat treatment, compared with unmodified alloy, there are more short rod-shaped and round borides in the modified alloy. Moreover, the impact toughness of Fe-B alloys containing cerium is higher than that of the unmodified alloy. The change of boride morphology from continuous network to disconnected network should be the main reason.

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