

# Effect of variable heat treatment modes on microstructures of Fe-Cr-B cast iron alloy

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**Abstract:** The effect of heat treatment mode on the microstructure of Fe-Cr-B cast iron alloys was investigated in this paper by comparing the difference of precipitation patterns of secondary particles after thermal cycling treatment (TCT) with those after normal heat treatment (NHT). No obvious differences were found in precipitation patterns of secondary particles between TCT and NHT when experimental temperature was below  $A_{r1}$ . However, when temperature was over  $A_{r1}$ , there were significant differences, with secondary particles prominently segregated at the grain boundaries under TCT, while the particles evenly distributed in the matrix under NHT. The reason for the microstructure differences could be associated with the development of non-equilibrium segregation of boron during TCT.

**Key words:** heat treatment mode; microstructures; Fe-Cr-B cast irons; non-equilibrium segregation of boron  
CLC number: TG143.9 Document Code: A Article ID: 1672-6421(2008)001-028-04

For some time, researchers have been attempting to develop ferrous wear-resistant alloys that can balance two significant properties, i.e., wear resistance and fracture toughness, because these two properties often contradict each other. For instance, steels, particularly low carbon steels, possess excellent ductility and fracture toughness, but have poor wear resistance owing to a lack of a high volume fraction of hard phases, such as borides, carbides or boron-carbides in the microstructures. Conversely, white cast irons, in particular, the white cast irons with high chromium, recognized as one of the best wear-resistant ferrous materials, have excellent wear resistance, but poor fracture toughness. This should be attributed to the existence of a high volume fraction (15 to 25 vol.%) of large hard eutectic  $M_7C_3$  carbides that occur in a continuous, three-dimensional network surrounding the dendritic matrix grains<sup>[1]</sup>. Recently, research at the University of Queensland has shown that the reduction of carbon content of the high chromium white cast irons from 3.2–3.5 wt% to 1.9 wt% led to significant improvements in fracture toughness from a value of 20 – 30 MPa · m<sup>1/2</sup> to ~ 40 MPa · m<sup>1/2</sup><sup>[2]</sup>. Therefore, the reduction of carbon content of high chromium cast irons is a promising solution to increase the toughness. However, the extent of this reduction is limited. If the carbon content is significantly reduced, the wear resistance can be markedly degraded due to the significant associated reduction in the volume fraction of the hard eutectic  $M_7C_3$  carbides. So

balancing the wear resistance and the fracture toughness by reducing the carbon content of the high chromium cast irons is still not an ideal approach.

The unique characteristics of boron in iron can provide an option to solve this problem. Since boron has limited solubility in iron, 0.004 – 0.008 at% at 900 °C and 0.0005 at% at 700 °C<sup>[3]</sup> and tends to bond with iron, chromium, vanadium, molybdenum, titanium etc, to form borides,  $M_2B$  or  $MB$ , which are just as hard or harder than the  $M_7C_3$  eutectic carbides ( $HV_{M_2B} = 1,200 - 1,700$ ,  $HV_{MB} = 2,000$ ,  $HV_{M_7C_3} = 1,200 - 1,600$ )<sup>[4-5]</sup>. Therefore, it should be possible to modify the high chromium white cast irons by substituting  $M_2B$  and  $MB$  borides for the eutectic  $M_7C_3$  carbides by high level of boron addition and essentially replacing most of the carbon in the alloys. The direct consequences envisaged for the high level of boron substitution of carbon of the high chromium cast irons are that the carbon contents, particularly in the iron matrix, would be drastically reduced. This should in turn result in improving the ductility and fracture toughness, while maintaining the inherently excellent wear resistance. The significance of this approach is that it allows the matrix and hard phases to be altered independently by varying carbon and boron contents. Lakeland developed this idea in 1991, and designed a group of Fe-Cr-B cast irons<sup>[6]</sup>, based on the composition of the high chromium cast iron. Concurrent with laboratory research, a series of field trials were carried out in Australia and in Sweden<sup>[6-7]</sup>. These field trials were only partially successful. The successful trials showed that the performance of Fe-Cr-B cast irons was superior to the nodular cast irons and to the high chromium white cast irons in the same component applications under the same conditions. While other trials

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Received: 2007-09-08; Accepted: 2008-01-20

demonstrated that, the alloys are sensitive to cracking, often leading to instant or premature failure. This is a direct consequence of the lack of detailed microstructure information. Therefore, a comprehensive understanding of the microstructures is crucial for the further development of Fe-Cr-B cast iron alloys. This paper presents a part of the research work, introducing the investigation on the effect of variable heat treatment modes on microstructure of Fe-Cr-B cast iron alloy.

### 1 Materials and methods

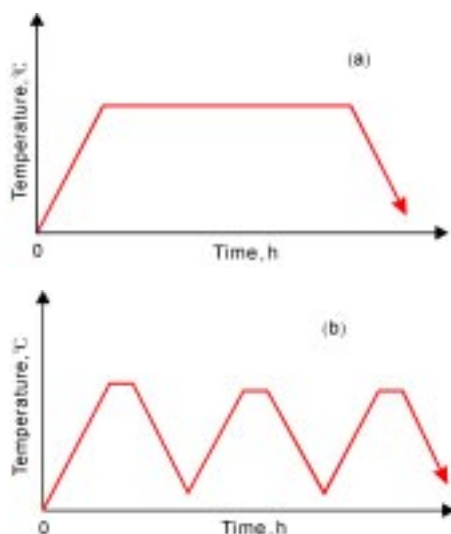
The typical composition of an experimental Fe-Cr-B cast iron is listed in Table 1. The cast iron was melted in an electric induction furnace with a magnesite lining and cast in “Y” sample moulds following ASTM A 781/A 781M-95. The test specimens were disks with dimensions of 20 mm diameter and 8 mm thickness, and were cut from the lower part of the “Y” blocks. The specimen surface was ground to remove 3 mm from the surface and thus eliminated any oxidized layer.

**Table 1 Composition of the experimental Fe-Cr-B cast iron alloy, wt%**

C	B	Si	Mn	Cr	Mo	V	Ni	Cu
0.164	1.4	1.16	0.24	11.3	0.88	0.66	1.1	0.55

Two different heat treatment modes, thermal cycling treatment (TCT) and normal heat treatment (NHT), were adopted as schematically shown in Fig. 1. Both treatments were carried out in an electric resistance furnace, which allowed automatic control of heating, quenching and cycle time. The thermal cycling treatment was for 17 min heating and soaking, followed by 2 min in coolants. The heat-through time in the experimental disks were measured about 6 min and the coolants used were 15 °C flowing tap water and air respectively.

The microanalysis was carried out by using microscope observation and x-ray diffraction. For the small particle analysis, carbon extraction replicas were used. The replicas were stripped electrolytically from the substrate in a 10 vol% nital solution. In

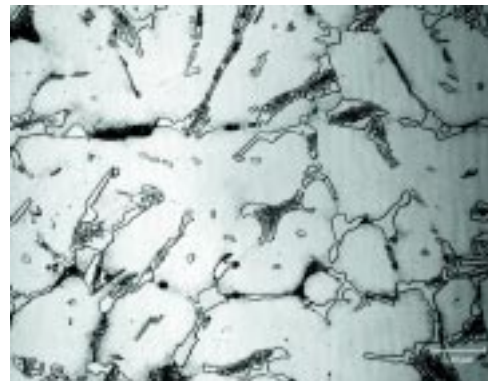


**Fig. 1 Schematic diagrams of the normal heat treatment (NHT) (a) and the thermal cycling treatment (TCT) (b)**

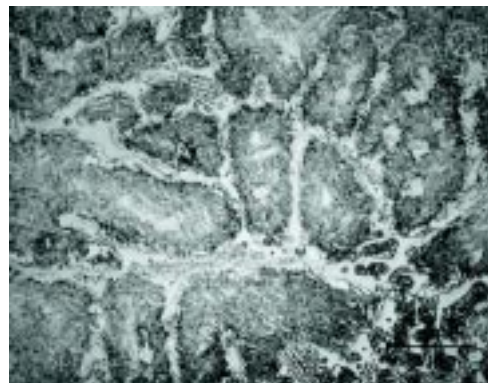
addition, dilatometer tests were carried out on a Dilatometer-DIL 402C, the 5 °C/min heating and cooling rates and argon gas were used for oxidation protection. The test temperature ranged from 50 °C to 1,050 °C. The specimens 5 mm × 5 mm × 25 mm for the dilatometer tests were also cut from the lower part of the “Y” blocks. These specimens were then ground into small cylinders 3 mm diameter × 10 mm long.

### 2 Results and discussion

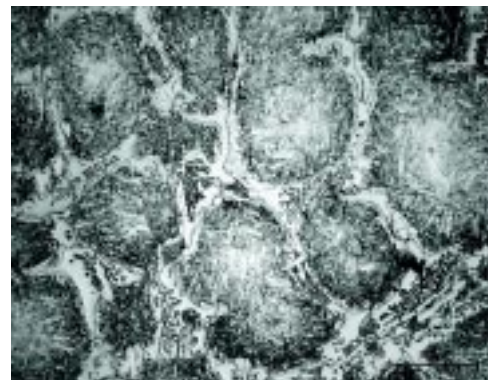
Figure 2 shows an as-cast microstructure of the experimental cast iron, comprised of a dendritic matrix with large interdendritic eutectic M<sub>2</sub>B borides [8]. At tempering temperatures over 500 °C, a high concentration of tiny particles started to precipitate from the matrix. These particles evenly distributed in the matrix and there were no apparent difference in tempered microstructures between TCT and NHT, as shown in Fig. 3. Increasing



**Fig. 2 As-cast microstructure of experimental cast iron**



(a) NHT at 650 °C for 90 min



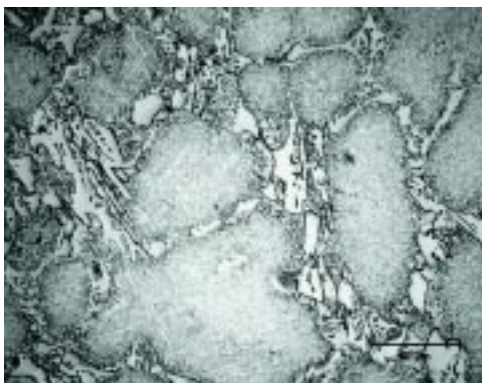
(b) TCT at 650 °C for 10 cycles

**Fig.3 The tempered microstructures of experimental alloy**

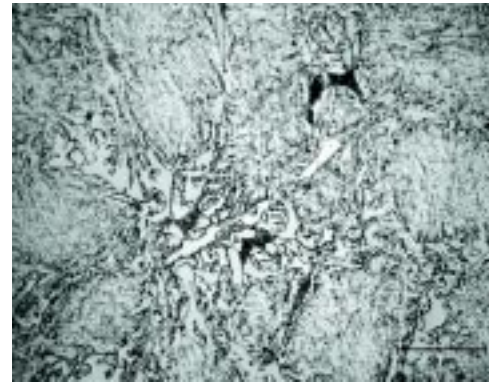
temperature and tempering time for NHT or the number of thermal cycles for TCT led to a coalescence of the tiny particles, but there was still less difference in the precipitation patterns in matrix between NHT and TCT.

Above 750 °C, the secondary precipitates in matrix after the TCT and NHT began to differ from each other. Figure 4 shows the micrographs subjected to (a) NHT at 850 °C for 90 min and (b) TCT at 850 °C for 10 cycles. Both treatments started at the as-cast state. It can be seen that the secondary particles precipitated at the sub-grain and grain boundaries were more pronounced under TCT, which formed acicular microstructures, as seen in Fig. 4(c) with magnified scale, compared with the secondary particles that were evenly distributed in the matrix under NHT. Increasing or decreasing soaking time at 850 °C under NHT, for instance, from 90 min to 150 min or from 90 min to 60 min, has less significant influence on the uniform distribution of the secondary particles in the matrix. But increasing or decreasing cycle numbers at 850 °C under TCT has a significant influence on the uneven distribution of the secondary particles. For example, when the number of cycles was increased from 10 to 15, the acicular microstructures were more prominent because more secondary particles segregated at the sub-grain boundaries. However, when the cyclic number was reduced from 10 to 5, the acicular microstructures, i.e., the segregation of the secondary particles at the sub-grain and grain boundaries was correspondingly reduced and the microstructure difference between TCT and NHT was also reduced. Therefore, the acicular microstructures at 850 °C under TCT are more closely associated with the cyclic number. Increasing the number of thermal cycles favors the formation of acicular microstructures. Apart from temperature and cyclic number, cooling rate also has significant influence on the microstructures of the experimental cast iron alloy. Decreasing cooling rate favors the formation of acicular microstructures in the matrix. For example, cooling in air after austenitizing at 850 °C under TCT led to a segregation of more secondary particles in sub-grain boundaries, compared with that cooling in water under similar TCT condition.

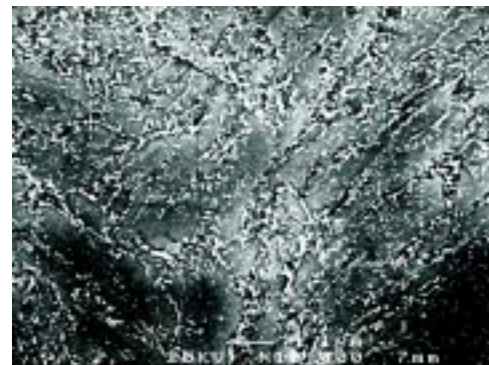
The identification of the particles in carbon extraction replicas excluded the possibility of the microstructure differences between Fig. 4(a) and (b) associated with the difference of the secondary particles. The secondary particles in both cases were identified as mixtures of different types of boron-carbides, such as  $M_{23}(C, B)_6$



(a) NHT at 850 °C for 90 min



(b) TCT at 850 °C for 10 cycles

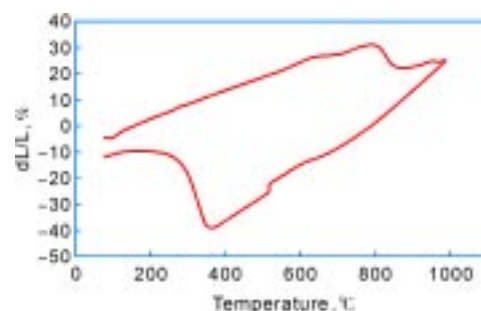


(c) Magnified matrix in (b)

**Fig. 4 The microstructures of experimental alloy after different treatment**

and  $M_6(C, B)$ , where M stands for the metallic elements, Fe, Cr, Mo and V [8].

Dilatometer tests indicated that austenite began to form at about 780 °C ( $A_{r1}$ ) and completed at about 900 °C, see Fig.5. So the formation of acicular microstructures or the segregation of the secondary particles in the sub-grain and grain boundaries subjected to TCT at 850 °C is associated with the formation of austenite, cooling rate and the number of thermal cycles.



**Fig. 5 Dilatometer curves of experimental alloy**

The segregation of the secondary particles at 850 °C subjected to TCT occurred during the heating and cooling processes, rather than the soaking process. For the case in Fig. 4, the total soaking time experienced in the two cases are nearly same, about 90 min, but their time of heating up and cooling down are considerably different. For the NHT case, the total heat treatment time are about 96.025 min, with about 6 min for heating up to the temperature, plus 90 min holding at the temperature and 0.025 min for cooling down in water. For the TCT case, the total accumulated heat treatment time during the 10 cycles are about

170 min, with a 60 min devoted to cumulative heating up plus a total of 90 min holding at the temperature and a cumulative 20 min for cooling in water. Thus, the TCT case experienced much longer heating up and cooling down terms than the NHT case. Therefore, it can be expected that it is the large difference of heating up and cooling down terms, rather than the soaking term between TCT and NHT that brought about the considerable difference in the distribution of the secondary particles.

The microstructure difference between TCT and NHT could be associated with the development of non-equilibrium segregation during TCT. From the mechanism of non-equilibrium segregation of boron at the grain boundaries, the development of the non-equilibrium process in boron alloys must meet the following two conditions<sup>[9-11]</sup>:

(1) Austenite must present in microstructures.

(2) There is vacancy flowing toward crystal defect in microstructures during quenching process.

Below 750 °C, there was only ferrite, and no austenite in matrix. So there was nothing of the non-equilibrium segregation of boron subjected to either TCT or NHT. Above 750 °C, austenite started to form in microstructures. So it can be expected that there was certain amount of austenite in matrix at 850 °C. In addition, the heating up and cooling down term during TCT promotes vacancy flowing. This could explain why the non-equilibrium segregation of boron occurred at 850 °C under TCT, and increasing the number of thermal cycles and decreasing cooling rate resulted in a segregation of more secondary phases at the sub-grain and grain boundaries.

It needs to point out that, besides the non-equilibrium segregation of boron to precipitate more secondary particles at the sub-grain and grain boundaries, the additional nucleation sites (short-lived) or an alternative growth mechanism during cycling processes might contribute to the segregation, too. This needs to conduct more research for the verification.

### 3 Conclusions

(1) Heat treatment parameters, such as temperature, cooling

rate and the number of thermal cycles, have significant influence on the microstructures of Fe-Cr-B cast iron alloy. When the temperature is over  $A_{r1}$  and austenites existed in the microstructures, both of increasing the number of thermal cycles and decreasing cooling rate will favor to develop the uneven distribution of the secondary particles in sub-grain and grain boundaries.

(2) The microstructure difference between TCT and NHT in Fe-Cr-B cast iron alloy could be associated with the development of the non-equilibrium segregation of boron to sub-grain and grain boundaries during TCT.

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