Effect of pre-annealing heat treatment on destabilization behavior of 28 wt. % Cr-2.6 wt. % C high-chromium cast iron

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Abstract. In the present work, the effects of pre-annealing heat treatment on microstructure and hardness in destabilized 28 wt. % Cr-2.6 wt. % C high-chromium irons have been investigated by X-ray diffraction, light microscopy, and scanning electron microscopy. As-cast specimens were annealed at 800°C for 4 h, and then furnace cooled. Destabilization after annealing was carried out at 1000°C for 2 and 4 h, followed by forced air-cooling to room temperature. Vickers macro-hardness was measured on specimens from each condition. The results revealed that the as-cast microstructure consisted of primary austenite dendrites with an interdendritic eutectic structure of M7C3 carbide and eutectic austenite. The eutectic austenite had partially transformed to martensite during cooling in the mold. During annealing at 800°C, austenite converted to carbides + ferrite and some pearlite. The hardness decreased from 507 to 425 HV30. Destabilization led to precipitation of secondary carbides and transformation of dendritic and eutectic austenite to martensite with some retained austenite. After destabilization, the macro-hardness increased from 736 HV30 in the iron without prior annealing up to 843 HV30 in the iron with annealing plus destabilization. For maximum hardness, prior annealing reduced the destabilization time from 4 to 2 h.

1. Introduction

High-chromium cast irons are widely used in applications in which high abrasive wear resistance is required such as wear parts in mining, mineral and cement industries. The performance of these irons can be improved by heat treatment, and alloying element [1-7] and their wear resistance depends on the type and the volumetric fraction of matrix and carbides. Although un-machined high-chromium cast iron casting can be used for abrasion resistance. There are some applications of these irons where machining operations become necessary such as the mating faces between pump bodies and their end liners and the faces of roll segments used in crushing rolls. Machined fitting faces ensure that correct assembly with other parts can be achieved during the production or maintenance of crushing and pumping equipment. High-chromium cast irons are difficult to machine due to the presence of large amounts of hard eutectic carbides coupled with work hardening austenitic or hard martensitic matrices in their microstructures. Machining such structures can cause rapid wear damage to cutting tools. To allow conventional tool machining in place of grinding, annealing heat treatments at 700-950°C can be used to reduce hardness levels to below 400 HV [8-12]. During annealing, austenite decomposes into ferrite and pearlitic and/or secondary carbides [8, 10-12]. The problems involved in the machining of white alloy irons, in general, have been considerably reduced by the use of cubic boron nitride (CBN)
cutting tool tips, which can replace conventional carbide or alumina ceramic tooling and grinding with silicon carbide wheels [13-14]. CBN tooling enables cost-effective machining of the as-cast austenitic matrix or hardened irons since it avoids the need to use grinding, which is not only slow but may also introduce grinding cracks. Even when CBN tooling is available annealing to produce lower hardness levels is often required notably when integral screw threads have to be provided to avoid the use of cast-in inserts in slurry pump parts [15]. After annealing treatment and machining, irons can then be hardened by re-austenitization and destabilization following by air hardening heat treatment to provide a martensite matrix with higher hardness and wear resistance. Destabilization involves heating to and holding at 900-1025°C for up to 6 h to destabilize the austenite matrix by encouraging precipitation of secondary carbides. After destabilization and air hardening, the microstructure consists of eutectic carbide (M6C3) and secondary carbides (normally M7C3 and/or M23C6) in a matrix of martensite and some retained austenite [1-4]. The hardness can be increased up to 800 HV30 after destabilization [3-4]. Irons are typically tempered after air hardening to transform retained austenite, reduce internal stress, and control final hardness to provide an optimum balance of wear and impact resistance for a given application [9-12, 16-17].

Apart from reducing the hardness of the irons to accommodate tool machining as mentioned earlier [8-12], pre-annealing was done before destabilization for homogenizing [17]. It was found that pre-annealing did no effect to the hardness of the irons after hardening, but it reduced the destabilization time to develop full hardness [17]. However, higher total hardness and lower destabilization time to establish full hardness were reported for the irons after destabilization with pre-annealing [12]. In the present work, the effects of pre-annealing at 800°C on the microstructure and hardness after destabilization of an iron-containing 28 wt. % Cr-2.6 wt. % C were investigated.

2. Experimental method

2.1. Material preparation and heat treatment

Table 1 shows the chemical composition for high-chromium cast iron prepared by melting in a magnesia crucible, using an electric resistance furnace. The metal was cast into a sand mold to produce cylindrical test bars with a diameter of 25 mm and length of 300 mm.

The as-cast samples were annealed at 800°C for 4 h in an electric furnace with a heating rate of 5°/min and then cooled in the furnace with a cooling rate of 40°/hr. Destabilization after annealing was performed at 1000°C for 2 and 4 h, followed by air-cooling to room temperature. This iron is air-hardenable.

| Element | Cr | C | Mo | Si | Mn | S | Ni | P | W | Fe |
|---------|----|---|----|----|----|---|----|---|---|----|
|         | 27.86 | 2.63 | 0.03 | 0.20 | 0.11 | 0.01 | 0.20 | 0.02 | 0.01 | Bal. |

2.2. Characterization and hardness testing

For microstructural examination, the samples were cut from the as-cast and heat-treated materials. Samples for X-ray diffraction technique (XRD) and scanning electron microscopy (SEM) were ground on silicon carbide papers and then polished with diamond paste. Specimens were etched in 50 ml of HCl and 10 g of Na2S2O3 in 100 ml distilled water. The microstructures were studied using an LEO 1455VP SEM operated at 20 kV. The phases present were determined using a Philips X’pert diffractometer with a Cu Kα X-ray source. Vickers macro-hardness testing was performed on un-etched specimens using 30 kgf load and 15 s indenting time. The mean values are based on eight different areas in each specimen.
3. Results and Discussion

3.1. Microstructures

The as-cast microstructures shown in figure 1(a), consisted of primary austenite ($\gamma$) dendrites with an interdendritic eutectic structure of $M_7C_3$ carbide ($M = Fe, Cr, Mo$) + eutectic austenite which was partially transformed to martensite ($\alpha'$) during cooling in the sand mold. After annealing at 800$^\circ$C for 4 h, as seen in figure 1(b), the ferrite ($\alpha$) matrix + secondary carbides were observed, due to decomposition of austenite during annealing. Small areas of pearlite were also present in the central regions of dendrite arms due to microsegregation effects. The presence of pearlite indicated that the transformation of austenite to ferrite and secondary carbides was incomplete [8].

![Figure 1](image1.png)

**Figure 1.** The microstructure of 28 wt. % Cr-2.6 wt. % C iron: (a) as-cast condition (b) annealing at 800$^\circ$C for 4 h.

![Figure 2](image2.png)

**Figure 2.** XRD traces obtained from 28 wt. % Cr-2.6 wt. % C iron: (a) as-cast condition (b) annealing at 800$^\circ$C for 4 h.
Figure 2 shows the XRD patterns of the as-cast iron and after annealing at 800°C for 4 h as compared with the XRD lines of austenite, ferrite, martensite, and M₇C₃ carbide. XRD results confirmed the presence of austenite, martensite and M₇C₃ carbide in the as-cast condition. Peaks from austenite at 2θ = 43.279°, 50.408°, 74.054°, martensite at 2θ = 65.542°, 81.943°, and M₇C₃ at 2θ = 39.135°, 44.370°, 50.375°, 52.553°, were observed. After annealing, M₇C₃ carbide and ferrite formed. Peaks from ferrite at 2θ = 44.674°, 65.023°, 82.335°, and 98.949°, were observed. The absence of some M₇C₃ peaks in the annealed iron can be attributed to texture effects.

![XRD patterns of iron](image)

Figure 3. The microstructure of 28 wt. % Cr-2.6 wt. % C iron: (a) after destabilization for 4 h, (b) pre-annealing plus destabilization for 2 h and (c) pre-annealing plus destabilization for 4 h.

The microstructures after destabilization for 4 h and annealing plus destabilization for 2 and 4 h, as shown in figure 3, consisted of secondary carbides in a martensite matrix. Pre-annealing increased the amount and sizes of secondary carbides after destabilization. At longer destabilization time of 4 h, as shown in figure 3(c), the secondary carbides were observed to be coarser and fewer in number. This is in agreement to previous work [3-4, 12] and can result from coarsening or the Ostwald ripening of the secondary carbide particles [3].

3.2. Macro-hardness
Figure 4 shows the effect of annealing and destabilization on the macro-hardness. After annealing at 800°C for 4 h, the macro-hardness decreased from 507 HV30 in the as-cast condition to 425 HV30. During annealing at the 800°C transformation of the austenite matrix to ferrite plus secondary and pearlitic carbides is expected to be almost complete such that the furnace cooling rate used in work gave an acceptable low hardness of 425 HV30 since no martensite was formed.

The macro-hardness after destabilization at 1000°C for 4 h in the iron without pre-annealing was 736 HV30. Pre-annealing increased the macro-hardness after destabilization at 1000°C for 2 and 4 h up to 843 and 830 HV30, respectively. This is most likely due to more even distribution or higher degree of homogenization of C and Cr as suggested by Inthidech et al. [17], and hence more uniform secondary carbide precipitation. Therefore, it can be suggested that pre-annealing affects increasing the hardness of the irons after destabilization hardening as reported by Cubillos et al. [12] and also
reduces the destabilization time to develop full hardness as reported by Cubillos et al. [12] and Inthidech et al. [17].

![Image](image.jpg)

**Figure 4.** Effect of annealing and annealing plus destabilization on the macro-hardness (AC = as-cast, AN = annealing, D4h = destabilization for 4 h, AN+D2h = annealing plus destabilization for 2 h, AN+D4h = annealing plus destabilization for 4 h).

4. Conclusions
The results obtained and their discussion make it possible to draw the following conclusions:

✓ The microstructure of the as-cast 28 wt. % Cr-2.6 wt. % C iron consisted of austenite dendrites, eutectic carbide and some martensite adjacent to carbides.

✓ The average as-cast hardness is 500 HV30. A hardness of 425 HV30 can be obtained after annealing at 800°C for 4 h.

✓ Pre-annealing before destabilization increased the amount and sizes of secondary carbides in the iron after destabilization.

✓ Pre-annealing as an effect on increasing the hardness of the irons after destabilization hardening. The maximum hardness of 843 HV30 was obtained in the iron with pre-annealing at 800°C for 4 h plus destabilization at 1000°C for 2 h.

✓ Annealing is used to facilitate machining. This study suggests that pre-annealing can also reduce the destabilization time needed to obtain the maximum hardness.

Acknowledgments
The Naresuan University, Thailand, is acknowledged for the financial support of this work under Grant No. R2562B078.

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