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Wear Behavior and Microstructure Changes of a High Chromium Cast Iron: The Combined Effect of Heat Treatment and Alloying Elements

Rim Chaima Touhami¹ · Said Mechachti¹ · Khedidja Bouhamla² · Ali Hadji¹ · Abdelkader Khettache¹

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Abstract

The microstructure and wear behavior of a manganese and niobium alloyed high chromium cast iron were examined and compared to an as-cast chromium cast iron. Three heat treatments were applied: quenching at 930, 980 and 1030 °C followed by tempering at 250 °C. The microstructures were characterized by optical and scanning electron microscopy equipped with an energy dispersive spectrometer. The Vickers microhardness (HV) were measured. X-ray diffraction tests were carried out in order to identify the nature of the existing crystallographic phases in each studied sample. The tribological behavior was studied in the dry state under a load of 10 N at room temperature. The friction coefficient was recorded. The wear mechanism of the investigated is observed by optical microscopy. The obtained results show that the as-cast microstructure of the studied chromium cast iron samples consists of an austenitic matrix and a complex eutectic carbide network of M_7C_3 type. The applied heat treatments have affected the microstructure and have promoted the formation of martensite, retained austenite and secondary precipitations. The addition of niobium and manganese also shows a refinement on the microstructure, this effect is observed on the dimensions of the dendrites which appear finer in the chromium cast iron alloyed with Mn and Nb.

Keyword High chromium cast iron · Wear · Alloying elements · Microstructure · Heat treatment

Introduction

High chromium cast irons (HCCI) are wear resistant materials used in various industrial applications, such as mining, siderurgical and cement industries [1, 2]. These materials are ferrous alloys containing 10 to 30% Cr and 2 to 3.5% C [3, 4]. Their microstructure consists of an austenitic or martensitic matrix with some retained austenite depending on their solidification and the adopted heat treatment, with the presence of M_7C_3 and $M_{23}C_6$ eutectic carbides type. These cast irons solidify as primary austenite dendrites with netted eutectic carbides and, during cooling, a part of the austenite turns into martensite. Typically, the castings undergo a quenching treatment followed by a tempering treatment to increase wear and impact resistance [5–7].

Rim Chaima Touhami touhami.rym23@gmail.com In previous research works, chromium cast iron was the subject of various research works intended to improve its mechanical properties [8–11]. To meet this objective, several methods have been used: (1) the addition of chemical elements [12–14]; (2) hardening using heat treatment or cryogenic treatment method [15–17]; (3) Dynamic-technique aiming at grain refinement by applying ultrasonic wave or mechanical [10–18].

The addition of alloying elements and the use of heat treatments is the most widely applied hardening methods for this type of cast iron because of their low production cost and the ease of handling when compared to other methods.

Numerous research works were conducted to achieve improved wear resistance of HCCI through the addition of various alloying elements such as vanadium, tungsten, niobium, molybdenum and titanium [12–14, 19]. During solidification, these carbide-forming elements react with carbon to form their own carbides or other complex carbides. In turn, the presence of these carbides contributes to the refinement of the microstructure and consequently improves the hardness and tribological behavior of the chromium alloyed cast iron [20–22]. However, many studies have pointed out that Mn stabilizes the austenite phase and adversely affects the

¹ Foundry Laboratory, BADJI Mokhtar University Annaba, BP 12, 23000 Annaba, Algeria

² Research Centre in Industrial Technologies – CRTI, P.O. Box 64, 16014 Cheraga, Algiers, Algeria

average hardness of the matrix, thereby reducing the wear resistance of materials. The Mn content in HCCI should be limited to less than 1 wt.% [8, 23]. Mn does not form its own carbide because it inserts into the M_7C_3 carbide and also dissolves in the matrix.

The application of heat treatments has also been proposed in order to obtain a martensitic structure with high secondary precipitation. The heat treatment commonly applied for maximum strengthening is quenching, which consists of heating the cast iron to temperatures between 800 and 1100 °C, followed by tempering in air at room temperature at temperatures between 200 and 600 °C [24].

The objective of this study is to investigate the effect of Manganese as well as Niobium and heat treatment on the microstructural changes, hardness and tribological behavior of high chromium cast iron. The recommended heat treatments were conducted to obtain a mixed structure with improved hardness and enhanced wear behavior.

Materials and Experimental Procedure

The basic high chromium cast iron is manufactured in an industrial induction furnace. The carbide-forming elements, namely manganese and niobium, were previously ground and weighed and then introduced into the melt. These ferroalloys were added as an ultra-fine powder in order to facilitate their dissolution in the metal liquid. The alloys were produced in furan resin sand molds as cylindrical specimens measuring 100 mm in length and 20 mm in diameter. The specimens obtained were cut under lubricant in view of getting a sufficient number of samples to conduct the different characterizations planned in this work. The chemical composition analysis was performed by spectrometry.

The heat treatment cycle considered in this work consists of a 3 h holding time hardening at different temperatures

Table 1	Wear	conditions
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Against	Normal load, N	Sliding speed, mm/S	Sliding distance, m	Tempera- ture	Time, min
Al ₂ O ₃	10	2	20	Room temper- ature	20

(930, 980 and 1030 °C) with forced air cooling. The samples were then tempered at 250 °C for 2 h with subsequent cooling in the furnace. The samples intended for metallographic characterization were prepared according to the conventional polishing procedure and then etched with 4% nital. The micrographic observations were carried out on an NIS-Elements D optical microscope and a JEOL JSM-6610LA SEM microscopes combined with EDAX. X-ray diffraction (XRD) spectra were recorded on a Rigaku diffractometer with Cu K α radiation ($\lambda = 1.5406 \text{ A}^{\circ}$) at 2θ and a step size of 0.02° in the range of 0 to 100° . The fitting of the diffractograms was carried out using X'Pert HighScor software in order to qualitatively assess the nature of the resulting microstructural phases. Hardness HV tests are carried on Zwick/Roelle Zhu with a load of 100 gf. The wear tests were carried out on a CSM Instruments tribometer. The applied conditions are presented in Table 1.

Results and Discussion

The chemical composition analysis of the studied chromium cast iron samples is presented in (Table 2). Based on the research results reported by Ping et al. [8] and Lu et al. [25], the eutecticity of the studied chromium cast iron is estimated by using following formula:

$$[\%C] + 0.0474 \times [\%Cr] = 4.3 \tag{1}$$

As shown by formula 1, the cast iron is hypereutectic if its eutecticity is higher than 4.3. In the studied case, the chromium cast iron exhibits an eutecticity lower than 4.3, meaning that it presents a hypoeutectic composition (Table 3). This type of cast iron solidifies according to the Fe-Cr-C system. It's as-cast microstructure consists of an austenitic matrix and a carbide network [1]. It is a wear resistant material intended for applications that are subject to abrasion, impact and friction.

Microstructure

As Cast

The optical and SEM micrographs obtained on the different as-cast (A and A1) and heat treated basic and alloyed high chromium cast iron are presented in Figs. 1 and 2

 Table 2
 Chemical compositions of the studied chromium cast iron (wt%)

Name	%C	%Cr	%Mn	%Mo	%Nb	%S	%P	Fe
A	2.07	14.82	0.54	0.07		0.07	0.05	Balance
A1	2.02	14.7	1	0.07	0.3	0.07	0.06	Balance

respectively. Based on the Fe-Cr-Cr diagram, the microstructure of this type of cast iron, in the as cast state, consists of a netted eutectic carbide embedded in an austenitic matrix [26]. These microstructures exhibit a hypoeutectic composition highlighted by the shape of the eutectic carbides and the primary austenite dendrites (Fig. 1a, b).

The addition of niobium and manganese at 0.3 and 1% respectively shows a refining effect on the size of the dendrites. The latter appear more refined in the Mn and Nb alloyed high chromium cast iron (Figs. 1b, 2b). Niobium appears in its pure form and does not dissolve in the matrix [27]. Filipovic et al. [28] indicate that the formation of NbC carbides limits the growth of primary dendrites in the austenite. The presence of niobium, which is more carburigenic than manganese, forms its own MC-type carbide [29, 30] which is known as a proeutectic carbide that precipitates at high temperatures while the metal is in the liquid state [31].

In addition, a high content of Cr and C and a relatively low content of Mn was detected at point 2, showing the existence of M_7C_3 type complex carbide. Since manganese and chromium have an atomic radius close to that of iron

Table 3 The $[\% C] + 0.0474 \times [\% Cr]$ of samples (wt.%)

Samples	%C	%Cr	[% C]+0.0474×[% Cr]	Structure
A	2.07	14.82	2.772	Hypoeutectic
A1	2.02	14.7	2.716	Hypoeutectic

[32], they occupy a substitutional position in the crystal lattice of the resulting carbide. Manganese is a carbide-forming element; it does not form its own carbide. It dissolves in the austenite up to its solubility limit and the remaining part contributes to the precipitation of M_7C_3 type carbides (Table 4) point 1-2 [8] (Fig. 3).

X-ray Diffraction Results

The refinement of the X-ray spectra of the basic and chromium alloyed cast iron with 0.3% niobium and 1% manganese in the as-cast state is shown in Fig. 4. The X-ray diffraction spectra reveal the presence of intense peaks of austenite in addition to martensite and M_7C_3 carbide. In the as-cast state, both types of cast iron exhibit a dominantly austenitic matrix. The niobium and manganese alloyed cast iron show the presence of niobium carbide. Considering the lower percentage of incorporated niobium, the proportion of carbide that can precipitate must obviously be reduced.

Heat Treatment Effect on Microstructure

The commonly applied heat treatment consists of holding at temperatures typically comprised between 800 and 1100 °C with a holding time of 1 up to 6 h. It causes a precipitation of secondary carbides. This precipitation takes place in the matrix. The type of secondary carbides depends on the added alloying elements. As a result of the heat treatment, the matrix is largely transformed into martensite with the presence of a proportion of untransformed austenite called retained austenite [20]. Since quenching can generate quench cracks, a tempering treatment at



Fig. 1 As-cast iron optical micrographs of: (a) basic cast iron (A), (b) Mn and Nb alloyed cast iron (A1)

M₇C₃ carbide

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Fig. 2 As-cast high chromium cast iron SEM micrographs: (a) base iron (A), (b) Mn and Nb alloyed cast iron (A1)

Table 4EDS composition (wt.%) from region of A1 in Fig. 3 as cast

Point	%C	%Cr	%Mn	Fe
1	3.32	8.81	0.9	86.98
2	13	41.32	0.78	44.23



Fig. 3 EDS Analysis of as-cast high chromium Mn and Nb alloyed cast iron (A1) $% \left(A^{\prime}\right) =0$



Fig.4 X-ray diffraction refinement of as cast basic high chromium cast iron (A) and as cast niobium and manganese alloyed high chromium cast iron (A1)

250 °C for 2 h with cooling in the furnace is required as a solution. The reduced content of alloying elements in the austenitic matrix increases the $M_{\rm S}$ temperature, so that upon cooling to room temperature, the matrix is subject to change to martensite. The microstructure shows a matrix

consisting of a mixture of martensite, retained austenite, secondary carbides and eutectic carbides. These microstructures are visible on the optical micrographs (Fig. 5) but the details of the secondary carbides (size, Fig. 5 Optical micrographs of heat-treated high chromium cast iron: (a) basic cast iron (A) treated at 930 °C; (b) alloyed cast iron (A1) treated at 930 °C; (c) basic cast iron (A) treated at 980 °C; (d) alloyed cast iron (A1) treated at 980 °C; (e) basic cast iron (A) treated at 1030 °C; (f) alloyed cast iron (A1) treated at 1030 °C



homogeneity, distribution) are visible on the SEM micrographs (Fig. 6).

Figures 7 and 8 display the EDS analysis of the ascast high chromium iron (A) and the niobium and manganese alloyed high chromium iron (A1) heat treated at 980 °C respectively. The EDS analysis (Figs. 7 and 8) and Tables 5 and 6 provide information on the changes that occurred after the heat treatment. The eutectic carbides generally of type M_7C_3 are not affected by the heat treatment; they remain intact. The measurement points marked in Fig. 7 represent secondary precipitation (point 1), a eutectic carbide (point 2) and a matrix area with secondary precipitation. The analyses represented in Table 5 reveal high carbon and chromium contents (point 1 and 3), indicating the presence of complex carbides containing chromium and carbon. On the other hand, in Fig. 8, the locations pointed out by the analysis are related to a secondary precipitation (point 1), a eutectic carbide (point 2) and a matrix area with secondary precipitations. Table 6 provides additional information on the analyzed locations where the presence of niobium carbide is clearly confirmed (A1) (Fig. 8, point 2).

Fig. 6 SEM micrographs of heat-treated high chromium cast iron: (a) basic cast iron (A) treated at 930 °C; (b) alloyed cast iron (A1) treated at 930 °C; (c) basic cast iron (A) treated at 980 °C; (d) alloyed cast iron (A1) treated at 980 °C; (e) basic cast iron (A) treated at 1030 °C; (f) alloyed cast iron (A1) treated at 1030 °C



Hardness

In order to study the effect of the added elements and heat treatments on the strengthening of A1 cast iron, microhardness have been measured and compared to A cast iron.

The obtained microhardness (HV) values presented in Fig. 9, show that A1 high chromium cast iron is harder than A high chromium cast iron in the as-cast case. However, this property has been more improved under the influence of heat treatments. Hardness of A1 high chromium cast iron is increased from 426 HV in the as-cast case to 493 HV after heat treatment at 930 °C, to 613 HV after heat treatments at 980 °C and to 700 HV after heat treatment at 1030 °C.

The increase of microhardness in the as-cast state of A1 compared to A high chromium cast iron is due to the effect of the added elements. Niobium is a strong carbide forming element. Its addition promotes the formation of MC-type carbides. In this case, these carbides are called proeutectic. They are known for their high hardness, which contributes to an increase of the casting hardness. High



Fig.7 EDS analysis of as cast high chromium cast iron (A) heat treated at 980 $^{\circ}\mathrm{C}$



Fig.8 EDS analysis of niobium and manganese alloyed high chromium cast iron (A1) heat treated at 980 $^\circ C$

Table 5 EDS composition (wt.%) from region of Ain Fig. 7 treated at 980 $^{\circ}\mathrm{C}$

Point	%C	%Cr	%Mn	Fe
1	29.25	9.46	0.55	60.75
2	29.26	30.13	0.85	39.76
3	29.26	10.62	0.51	64.61

Table 6 EDS composition (wt.%) from region of A1 in Fig. 8 treated at 980 $^{\circ}\mathrm{C}$

Point	%C	%Cr	%Mn	Fe	%Nb
1	29.75	30.33	0.62	39.31	
2	28.04	6.84		45.29	19.82
3	23.61	8.96	0.54	66.88	



Fig. 9 Effect of the austenitization temperature on the microhardness evolution of the studied chromium cast irons

temperature austenitization followed by forced air cooling causes a transformation of austenite into martensite. The microhardness improvement obtained after heat treatment is explained by the formation of martensite and precipitation of secondary carbides. The microhardness values measured on the samples treated at 930, 980 and 1030 °C clearly show the effect of the applied heat treatment on the properties of the studied cast iron. A proportional relation between the heat treatment temperature and the measured hardness values is noted. This justifies an increased secondary precipitation induced by the austenitization temperature change.

Wear Resistance

Evolution of the Friction Coefficient

Figure 10 presents the variation of the friction coefficient for basic iron (A) (Fig. 10a) and alloyed iron (A1) (Fig. 10b) with different treatments 930, 980 and 1030 °C, as a function of the sliding distance with an applied load of 10N. These curves reveal two successive periods of friction. The first is the running-in period, in this period the coefficient increases rapidly to reach a maximum value. This period is characterized by significant wear and deformation of the stabilization of the friction coefficient whose value is maintained constant whatever the sliding distance, during this stage the solid wear debris (third body) is formed during the sliding process [33].

It can be noted that the increase of the austenitization temperature leads to a decrease of the friction coefficient for the basic (A) and the treated cast iron (A1) (Fig. 11).



Fig. 10 Coefficients of friction of: (a) the as-cast iron (A); (b) alloyed iron (A1)



Fig. 11 Relationship between the friction coefficient and austenitization temperature

The friction coefficients of the heat treated and alloyed cast iron (A1) are relatively lower than that obtained on the basic cast iron. This is at first a consequence of the applied heat treatment temperatures and secondly the added carbide forming elements (Mn and Nb). The temperature rises from 930 to 1030 °C contributed to an increase of the amount of martensite and secondary precipitations. The addition of Nb permitted the formation of NbC carbide which leads to a better friction coefficient and wear resistance [34].

Wear Tracks Morphology (2D Views of Wear Tracks)

The microscopic analysis of the wear tracks is required to understand the wear type produced on each cast iron. The worn traces obtained on the studied materials, under the normal force 10N and applied speed are clearly shown in Fig. 12a–h. Numerous grooves of different widths are obtained on the as cast high chromium cast iron and are well highlighted by the Fig. 12a, c, e, and g. In alloyed high chromium cast iron (A1) Fig. 12b, d, f, h, a decrease in the width of the wear tracks is noted. These grooves, can be attributed to an increase of the wear rate. In Fig. 12, wear debris are well highlited. This phenomenon inevitably generates a third body which is an obvious consequence of the surface process degradation [34].

The grooves parallely oriented to the sliding direction support the abrasion wear mechanism. The latter is induced by the hard particles (carbides) as by the wear



Fig. 12 Optical microscope images of worn surfaces: (a) A as cast; (b) A1 as cast; (c) A treated at 930 °C; (d) A1 treated at 930 °C; (e) A treated at 980 °C; (f) A1 treated at 980 °C; (g) A treated at 1030 °C; (h) A1 treated at 1030 °C

debris of the deformed area observed on the cast iron [29]. The effect of the addition of Mn and Nb on the wear behaviour of the studied samples austenitised at 930, 980 and 1030 °C show almost the same pattern, the change is due to the wear rate, which decreases with increasing temperature and the wear track, which decreases with increasing temperature. This could be attributed to the microstructure refinement effect, which corresponds to the mixed structure of chromium carbide and nibium carbide particles obtained by the addition of Nb [7].

Conclusion

The microstructure and tribological properties of high chromium cast iron alloyed with manganese and niobium were investigated. The main conclusions obtained are the following:

- 1. The microstructure of the basic high chromiuim cast iron containing 2%C and 14%Cr with hypoeutectic composition, consists of an austenitic matrix and a $((Fe,Cr)_7C_3)$ carbide network.
- 2. The addition of manganese and niobium at contents varying between 1 and 0.3% respectively provoked a refining effect on the microstructure, this effect is noticed on the dendrite size, that appears more refined in the Mn and Nb alloyed cast iron. The addition of low contents of strong carbide-formingelements such as Nb (0.3%) favor the formation NbC carbide.
- 3. The heat treated microstructure of the examined high chromium cast iron shows a matrix consisting of a mixture of martensite, retained austenite, secondary carbides and eutectic carbides.
- 4. The heat treatments and the addition of Mn and Nb improve the microhardness and the wear behavior of the studied cast iron. These properties also enhance with the increase of the heat treatment temperature.
- Both chromium cast iron (basic and alloyed) treated at 1030 °C has a low wear rate, compared to the other one treated at 930 and 980 °C which means a better tribological behavior.
- The abrasion resistance of the studied high chromium cast iron (14.82% Cr, 2.07% C and 0.54% Mn) can be improved by adding small proportions of Mn and Nb (0.48% and 0.3%) respectively and by austenitising at 1030 °C.

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