

## THE EFFECT VANADIUM CONTENT AND HEAT TREATMENT ON WEAR RESISTANCE AND FRACTURE TOUGHNESS OF FE-CR-C-V ALLOY

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### Abstract

Experimental results indicate that the volume fraction of the carbide phase, carbide size and distribution had an important influence on the wear resistance of Fe-Cr-C-V alloys under low-stress abrasion conditions. Besides, the martensitic or martensite-austenitic matrix microstructure more adequately reinforced the  $M_7C_3$  eutectic carbides, minimizing cracking and removal during wear, than did the austenitic matrix. The secondary carbides which precipitate in the matrix regions of high chromium iron also influence the abrasion behaviour. The results of fracture toughness tests show that the dynamic fracture toughness in Fe-Cr-C-V white cast irons is determined mainly by the properties of the matrix. The high chromium iron containing 1.19 wt.% V in the as-cast condition, showed the greater dynamic fracture toughness when compared to other experimental alloys. The higher fracture toughness was attributed to strengthening during fracture, since very fine secondary carbide particles were present mainly in an austenitic matrix. In heat treated Fe-Cr-C-V alloys with varying contents of vanadium, lower  $K_{Ic}$  values were obtained, compared with as-cast alloys.

*Key words: Fe-Cr-C-V alloys, vanadium content, carbides, matrix microstructure, hardness, wear resistance, fracture toughness*

### Introduction

High chromium white cast irons are an important class of wear resistance materials currently used in a variety of applications where stability in an aggressive environment is a principal requirement, including the mining and mineral processing, cement production, slurry pumping and pulp and paper manufacturing industries.

Most improvements in abrasion resistance are accompanied by decreased toughness, primarily due to the eutectic carbide content. Nonetheless, a range of properties can be obtained by matrix variations to obtain optimum combinations of wear

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resistance and resistance to premature failure; such changes can be achieved by alloying and/or heat treatment [1-5].

It is already known that many factors influence the abrasive wear resistance of a material. For a given system (abrasive type, load, etc.), the microstructure of alloy plays a significant role in its wear resistance [5-8].

The influence of the microstructure of the high-chromium white irons on their abrasion resistance has been studied extensively. Previous research has recognized that the abrasion resistance of these alloys is primarily determined by the features of  $M_7C_3$  carbides, such as their volume fraction [7,9-11], hardness [1,11], morphology [12,13] and orientation [14-17]. The structure of the matrix that supports the carbides may be extensively altered by alloying and heat treatment. It has been already shown that both factors have significant influence on the wear behaviour [7]. There are several reports studying the effect of the matrix phase on the abrasive wear resistance of the high-chromium white irons [5,10,18,19]. Based on the available literature, there is still some uncertainty about the effect of the matrix structure (austenitic or martensitic) on the maximum abrasion resistance [6,10,20,21]. Retained austenite reduces the bulk hardness and therefore might be expected to reduce abrasion resistance. Some authors have found, however, that retained austenite actually improves abrasion resistance, due to its work-hardening properties [2,22]. Nevertheless, it is supposed that a martensitic matrix (containing little retained austenite) performs better with soft abrasives, while an austenitic matrix gives the better wear resistance with hard abrasives [5,18]. More precisely, it has been found that for a given wear situation, the best abrasion resistance is obtained with a retained austenite content of greater than 20% [19,23]. Also, the secondary carbides play an important role in determining the wear resistance of high chromium white cast iron [3,4,23,24].

The majority of studies which examine the effect of microstructure on fracture toughness have considered the role of carbide volume and matrix structure. The eutectic carbides play a strong role in influencing the fracture toughness of white irons [7,25-27]. It has been observed that the proportion of eutectic carbides on fracture surfaces is greater than that measured on polished sections [25]. This reflects the brittle nature of the eutectic carbides, with cracks preferentially following the carbide structure. Increasing the carbide volume would increase the number of brittle carbides in the structure, while reducing the amount of matrix material between these carbides. As expected, increased carbide volume decreases the fracture toughness for both austenitic and martensitic matrix [9,25,27]. A number of researchers have found that alloys with an austenitic matrix possess higher fracture toughness than those with a martensitic matrix [7,9,10], and that as the amount of austenite in the matrix increased, so did the fracture toughness [11]. It is supposed that the higher energy required for crack initiation and the slower crack propagation of existing cracks in austenite are responsible for its superior toughness [9]. Strain induced martensite formation in austenitic alloys also improves the toughness, by reducing the energy available for cracking. Voids, often present on the fracture surface of both austenitic and martensitic alloy, are recognized around secondary carbides located in the matrix structure. These secondary carbides are reported to be detrimental to toughness [12,28].

Extensive industrial applications of high-chromium white cast irons have attracted researchers to try different carbide-forming elements such as tungsten [29,30], niobium [29,31,32], vanadium [3,23,29,32-40], titanium [45,31,41,42] and boron [8,16]

to further improve this type of material. The addition of an alloying element which confines carbon in the form of a carbide different from cementite, with a greater hardness and more favourable morphology, and which reduces the carbon content of the matrix, allows the simultaneous improvement of both toughness and abrasion resistance. By controlling the morphology of the carbide phase and the matrix structure in these materials, significant improvement of toughness and service life may be achieved. Vanadium appeared to be of special interest, due to its double effects, on both the matrix structure and stereological characteristics of carbides.

In the present work, the effect of the subcritical heat treatment on the microstructure of high chromium white cast irons alloyed with vanadium and the properties relevant to the service performance of these alloys, namely the abrasion resistance and toughness, has been explored.

### Experimental Procedure

The chemical composition of tested alloys is listed in Table 1. An induction furnace was used for melting, and 200 mm long and 30 mm diameter rods were cast in sand moulds. Samples for structural analysis, hardness, wear, and fracture toughness tests were cut from the cast rods.

Table 1. Chemical composition of Fe-C-Cr-V white irons.

Alloy	Chemical composition (mass.%)									
	C	P	S	Si	Mn	Mo	Cu	Ni	Cr	V
1	2.89	0.025	0.061	0.85	0.71	0.48	0.99	0.100	19.03	0.0012
2	2.92	0.026	0.063	0.87	0.76	0.45	1.02	0.099	19.01	0.12
3	2.88	0.025	0.061	0.86	0.72	0.42	0.98	0.098	18.89	0.49
4	2.92	0.025	0.061	0.85	0.75	0.43	1.01	0.098	19.04	1.19
5	2.87	0.024	0.063	0.87	0.73	0.44	1.01	0.099	18.92	2.02
6	2.91	0.027	0.061	0.84	0.73	0.44	1.00	0.096	19.05	3.28
7	2.93	0.026	0.062	0.83	0.74	0.43	1.01	0.098	19.07	4.73

Samples were heat treated in an electric furnace at 950°C for 1h and then were air cooled to room temperature.

The microstructure was examined using conventional optical microscopy (OM), scanning electron microscopy (SEM). Samples for optical microscope examination were prepared using standard metallographic technique (etched with picric acid solution (1g) in methanol (100 ml) by adding 5 ml of hydrochloric acid). The morphology of  $M_7C_3$  eutectic carbides was examined by a scanning electron microscope, JEOL 733-FXA, using an accelerating voltage of 25 kV. For this examination, the polished samples were deep etched in a 10% HCl solution in methanol for 24 h then cleaned in an ultrasonic bath.

Cr  $K_\alpha$  radiation has been used for measuring the amount of retained austenite by means of X-rays, as it is considered to be more appropriate for structures containing greater amounts of carbides (in order to increase the dispersion, if there are interference peaks, for example) [43]. A continuously rotating/tilting specimen holder was used to eliminate the effect of the preferred orientation of the columnar structure, which has been shown to affect the results (as it has been described in detail in Ref. 44). At a scanning rate of  $1^\circ \text{ min}^{-1}$ , the integrated intensities under the peaks (200) $\alpha$ , (220) $\alpha$ , (220) $\gamma$  and (331) $\gamma$  were measured by a diffractometer.

The hardness of nickel-chromium and high chromium white cast iron alloys was examined by the Vickers method at 294 N load.

Abrasive wear resistance was evaluated according to the ASTM Standard Practice G-65, Procedure B (Dry Sand/Rubber Wheel Abrasion Test) [45]. Rounded quartz grain sand of sizes 50–70 mesh was used as an abrasive particle. The volume loss,  $\Delta V$ , was then calculated by dividing mass loss by the alloy density. The reciprocal value of the volume loss,  $\Delta V$ , due to wear is called wear resistance,  $\Delta V^{-1}$ .

Dynamic fracture toughness was measured at room temperature using an impact test machine equipped with an instrumented Charpy tub. The testing methodology selected was based on the three-point bending tests. The specimens, of 10 mm  $\times$  10 mm  $\times$  55 mm in size, were notched and precracked by fatigue following ASTM E399 recommendations [46]. The dynamic stress intensity factor,  $K_{Id}$ , was determined using the following equation [47,48]:

$$K_{Id} = \left( \frac{P_{max} S}{BW^{3/2}} \right) f \left( \frac{a}{W} \right) \quad (1)$$

where  $P_{max}$  is the maximum load,  $S$  is the span,  $B$  is the specimen thickness,  $W$  is the specimen width,  $a$  is the initial crack length and  $f(a/W)$  is a geometry factor.

## Results

The microstructure of the tested alloys in as-cast conditions are shown in Fig. 1. In addition to primary austenite dendrites and eutectic composed of  $M_7C_3$  carbides and austenite,  $V_6C_5$  carbides were also present in the structure of Fe-Cr-C-V alloys [40].

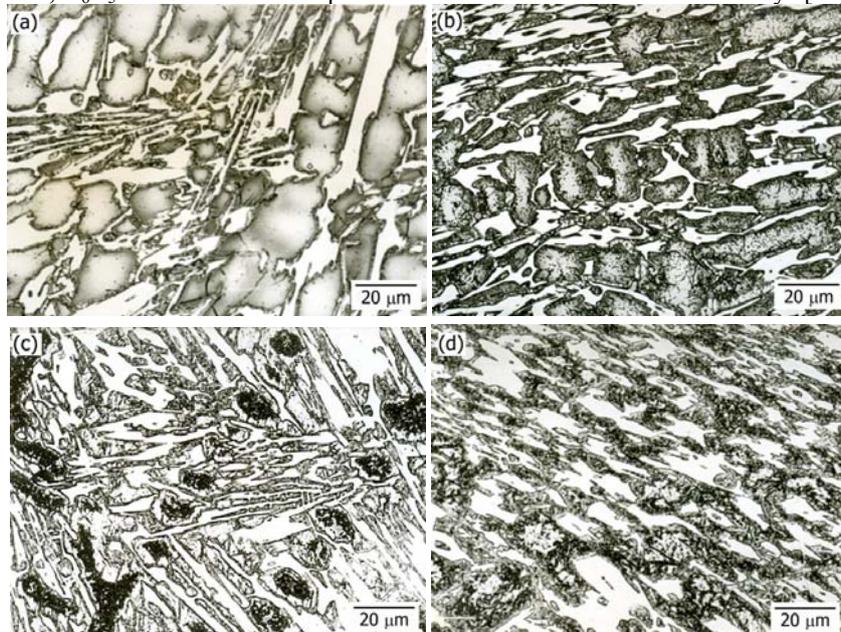


Fig. 1. Optical micrographs of Fe-Cr-C alloy (a) and Fe-Cr-C-V alloy containing 2.02% V (b), 3.28% V (c), 4.73% V (d) in the as-cast state.

The volume fraction and size of phases in the as-cast microstructure of the tested alloys are shown in Table 2. Vanadium influenced the refinement of the structure of high chromium white cast iron. The dendrite arms spacing (DAS) and size of eutectic  $M_7C_3$  carbides are decreased, while the size of  $M_6C_5$  carbides is increased with increasing vanadium content. In addition, with increasing vanadium content in the alloy, the volume fraction of primary austenite is decreased, whereas the amount of  $M_7C_3$  and  $M_6C_5$  carbides are increased.

Table 2: The volume fraction and size of phases in the microstructure of the tested Fe-Cr-C-V alloys.

alloy	V in alloy (mass%)	volume fraction (vol.%)			size ( $\mu\text{m}$ )		
		primary $\gamma\text{-Fe}$	$M_7C_3$	$M_6C_5$	DAS	$M_7C_3$	$M_6C_5$
1	-	50.83	30.97	-	14.08	7.48	-
4	1.19	47.48	31.96	1.58	12.76	6.74	1.26
5	2.02	45.76	32.82	2.31	11.93	6.52	1.31
6	3.28	42.05	34.31	3.12	10.67	5.67	1.45
7	4.73	39.15	35.47	4.27	9.45	5.03	1.52

The SEM micrographs of deeply etched samples revealed that single  $M_7C_3$  carbides in all the tested alloys were rod or blade shaped (Fig. 2). Vanadium, however, changed the morphology of eutectic colonies whose shapes depended mainly on the amount and shape of austenitic dendrites. A larger number of rods and blades usually formed sheaves in different directions. Sheaves of rods and blades interlaced among one another to form a continuous carbide network (Fig. 2a). In alloys containing 4.73% V, regions in which the border between carbide rod sheaves of different directions was clear, could also be observed (Fig. 2b).

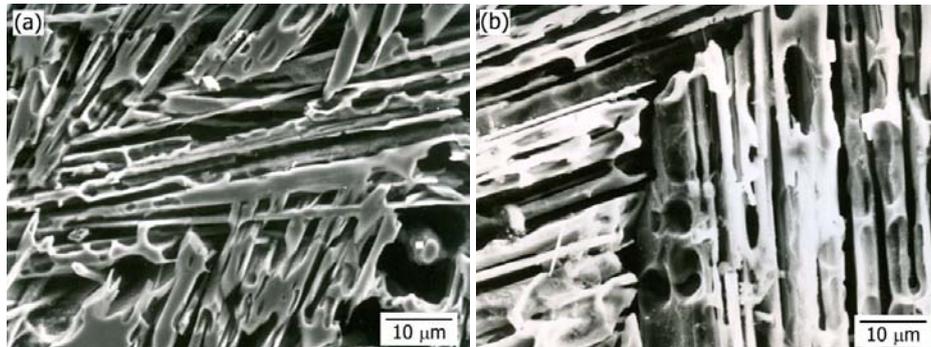


Fig. 2. SEM micrographs of deep etched sample showing morphology of  $M_7C_3$  carbides in Fe-Cr-C-V alloy containing: (a) 2.02% V and (b) 4.72% V.

The as-cast matrix microstructure of a basic Fe-Cr-C alloy is mainly austenitic. The inhomogeneity of the dendrites in Fe-Cr-C-V alloys containing 0.49% -4.73% V can be clearly seen (Figs. 1b-1d). Fine dark particles predominantly located in the middle zone of the austenite (Figs. 1b-1d) were identified to be carbides of  $M_{23}C_6$  type [39].

As it can be seen from Figs. 1a and 1b, in alloys containing less than 3.28% V, primary and eutectic austenite is transformed in a narrow zone along the border with eutectic  $M_7C_3$  carbide. Nevertheless, in Fe-Cr-C-V alloys containing 3.28% V (Fig. 1c) and 4.73% V (Fig. 1d), a remarkably higher degree of austenite transformation can be seen. TEM analysis identified martensite as a product of this transformation [39].

Therefore, an increase in the concentration of vanadium in the alloy (Fig. 3) would reduce the volume fraction of retained austenite.

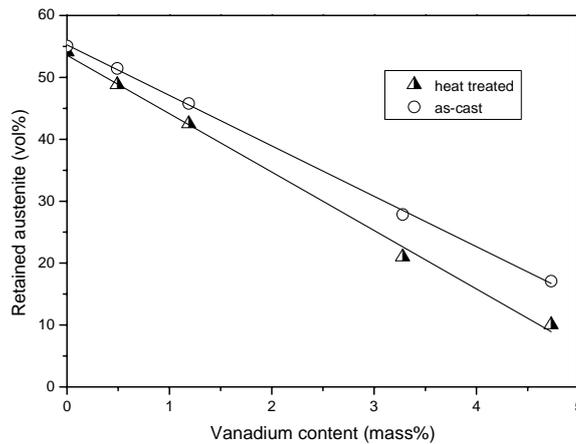
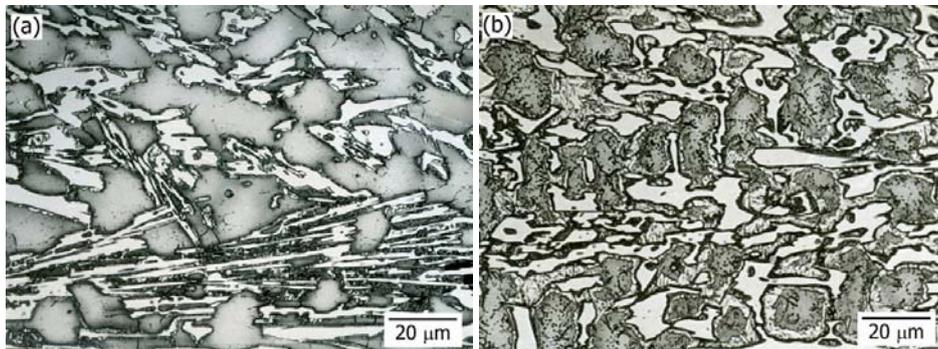


Fig. 3. Volume fraction of retained austenite as a function of vanadium content in the alloy.

The microstructure of tested Fe-Cr-C-V alloys in heat treated conditions is illustrated by the optical micrograph in Fig. 4. After heat treatment there were no notable changes in the structure of a basic Fe-Cr-C (Fig.4a). However, during isothermal holding at 500°C for 4 h, secondary carbides precipitated in the austenite of Fe-Cr-C-V tested alloys (Fig. 4b-4d). Martensite present in the as-cast structure was also tempered at this temperature. Austenite was then partly transformed into martensite during the cooling process. The amount of precipitated carbides and volume fraction of martensite which formed depended on the basic as-cast structure, i.e. on vanadium content in the alloy.



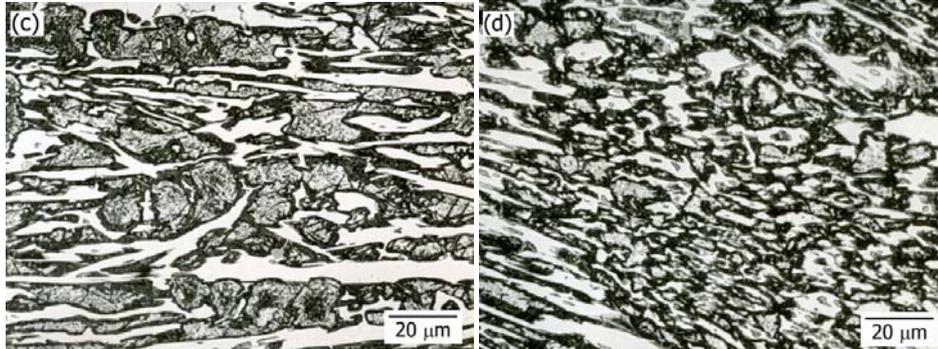


Fig. 4. Optical micrographs of Fe-Cr-C alloy (a) and Fe-Cr-C-V alloy containing 2.02% V (b), 3.28% V(c), 4.73% V (d) in the heat treated state (500°C/4h).

The matrix microhardness, and the alloy macrohardness, were improved by increasing the vanadium content in both the as-cast and heat treated state (Figs. 5 and 6).

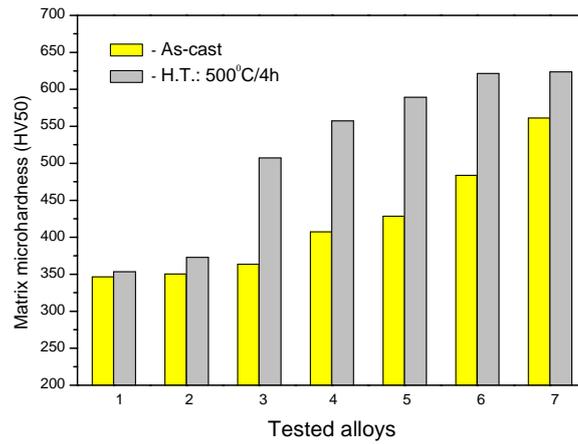


Fig. 5. Matrix microhardness in tested alloys.

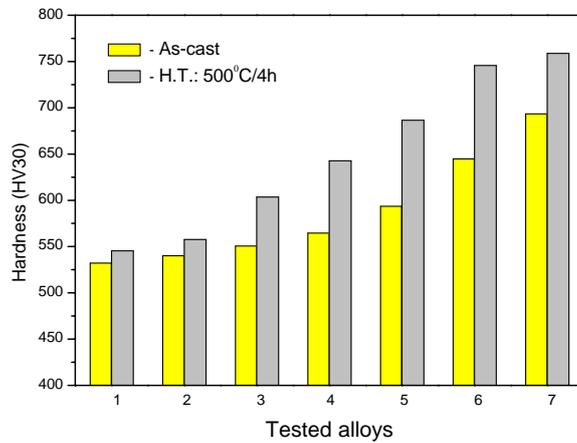


Fig. 6. Hardness in tested alloys.

The influence of vanadium on abrasion wear resistance is presented in Fig. 7. Wear resistance improved as the concentration of vanadium increased up to 3.28%, and fell off thereafter. Abrasion resistance was slightly higher after heat treatment at 500°C (Fig. 7).

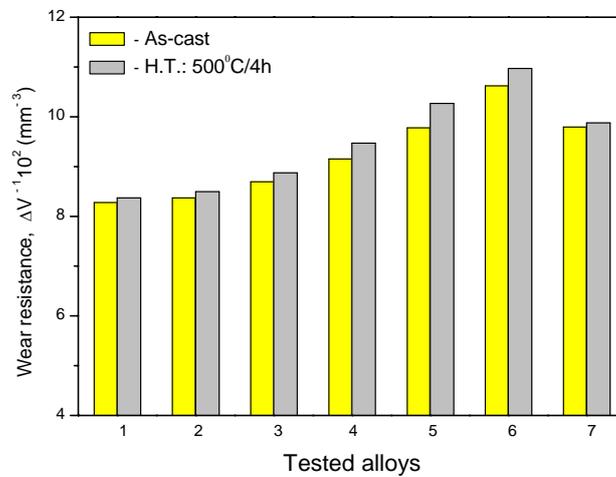


Fig. 7. Wear resistance in tested alloys

Vanadium contributes to the improvement of dynamic fracture toughness of Fe–Cr–C–V alloys in the as-cast condition. An alloy containing 1.19% V showed the highest fracture toughness (Fig. 8). However, in heat treated Fe–Cr–C–V alloys with varying contents of vanadium, lower  $K_{id}$  values were obtained, compared with as-cast alloys (Fig. 8).

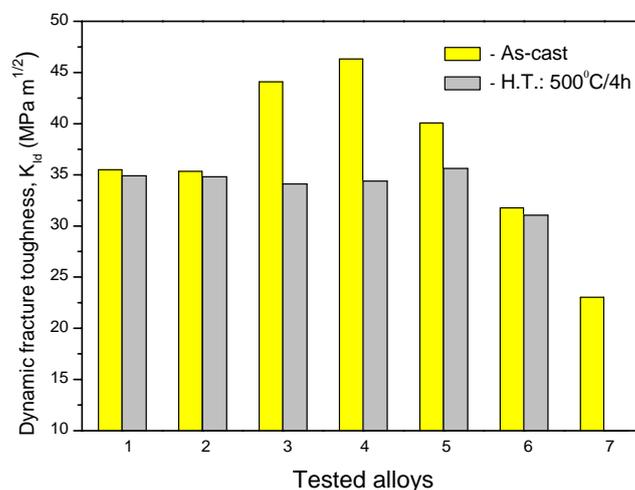


Fig. 8. Dynamic fracture toughness in tested alloys.

## Discussion

Vanadium affects the crystallization process in high chromium white irons. The changes in the volume fraction and size of the phases in the structure of Fe-Cr-C-V alloys (Table 2) indicate that with increased vanadium content, the alloy composition approaches the eutectic composition in the quaternary Fe-Cr-C-V system, causing a decrease of the solidification temperature interval.

The transformation of austenite to martensite in the Fe-Cr-C-V, as well as in the Fe-Cr-C white iron, is closely connected with the secondary carbide deposition. In the process of cooling after solidification, this transformation is possible only after destabilization of the austenite by the secondary carbide particles precipitation. The degree of martensitic transformation is determined by the amount of precipitated carbide, i.e., it depends on the austenite composition. This effect was considered previously in detail [39].

The improved hardness of the Fe-C-Cr-V white irons by increasing vanadium content in as-cast condition (Fig. 6) was the result of an increased volume fraction of the eutectic  $M_7C_3$  and  $V_6C_5$  carbide. Nevertheless, an increase in the amount of precipitated carbides in austenite and the formation of a larger amount of martensite, combined with a reduction in the volume fraction of retained austenite (Fig. 3), improved the matrix microhardness (Fig. 5), and consequently the alloy macrohardness (Fig. 6) in both the as-cast and heat treated states.

The abrasion resistance of the carbide phase was more effective than the matrix in white cast irons, since, mainly, the hardness of  $M_7C_3$  (1200-1800 HV) carbides were greater than the hardness of the used abrasive - quartz (900-1080 HV). In white cast irons the matrix was preferentially worn by a cutting action, under low-stress abrasion conditions. Eutectic carbides were then forced to stand in relief and directly obstruct the cutting action of abrasive particles for a period of time until they were partly or completely removed by a cutting or chipping action [7].

In other words, wear under low-stress abrasion conditions, and using a quartz abrasive, was apparently controlled by the rate of removal of the carbide phase since the protruding carbides protected the matrix from direct attack of abrasive particles.

Therefore, increasing the volume fraction of eutectic  $M_7C_3$  carbides up to 34% in the Fe-C-Cr-V tested alloys, and simultaneously increasing the amount of very hard  $V_6C_5$  carbide (2800 HV) reduced volume loss caused by abrasive wear (Fig. 7). However, a further increase in the volume fraction of the carbide phase (alloy with 4.73% V) (see Table 2) reduced wear resistance (Fig. 7), which was most likely attributable to intensive spalling of massive carbides during wear. A break in the continuity of the carbide network observed in this alloy (Fig. 2b) also contributed to reduced wear resistance.

In addition to the volume fraction of carbides, the size of phases present in the structure was another microstructure variable which affected the abrasive resistance of the Fe-C-Cr-V alloys. The smaller size of primary austenite dendrites (Table 2), i.e. the average distance between carbide particles, caused by increasing the content of vanadium in the alloy, protected the matrix better from direct attack by abrasive particles. The shorter rods of  $M_7C_3$  carbides (Table 2) also tended to be less severely cracked during wear, thereby improving the wear resistance of the alloys. It must be specified, however, that the effect of controlling wear rate by refining the microstructure depended on abrasive grit size, i.e. the ratio of mean free path to mean abrasive asperity size [9,21,35]. The smaller the ratio, the less likely that grit would undermine the hard particles by penetrating the matrix.

The improved wear resistance of white cast irons after heat treatment, compared with the as-cast condition, as shown in Fig. 7, indicated that wear resistance under low-stress abrasion conditions also depended on the matrix microstructure. In addition to the fact that the matrix helped control the penetration depth of abrasive particles, it also played an important role in preventing bodily removal of smaller carbides and cracking of massive ones [9,10]. Experimental results indicate that the martensitic or martensite-austenitic matrix microstructure more adequately reinforced  $M_7C_3$  eutectic carbides to minimize cracking and removal during wear under low-stress abrasion conditions than the austenitic matrix.

Besides, the secondary carbides which precipitate in the matrix regions of high chromium white irons also influence the abrasion behaviour. By increasing the matrix strength through a dispersion hardening effect, the fine secondary carbides can increase the mechanical support of the eutectic carbides. These results agree with those of Liu et al. [2] and Wang et al. [4] who found that the precipitation of fine  $M_{23}C_6$  carbides and the more homogeneous carbide distribution as a result of cryogenic treatment is responsible for the improved wear resistance of high chromium white irons.

The higher content of vanadium in high chromium white iron increases the volume fraction of the carbide phase and reduces fracture toughness. On the other hand, the toughness has been improved by reducing the size of  $M_7C_3$  eutectic carbide. However, the results of fracture toughness tests in both as-cast and heat treated conditions (Fig. 8) show that the dynamic fracture toughness in white cast irons is determined mainly by the properties of the matrix. Experimental results indicate that the austenite is more effective in this respect than martensite.

By increasing the vanadium content, the amount of retained austenite decreased, as it shown in Fig. 3, which subsequently reduced toughness. However, the Fe-C-Cr-V

alloys containing 1.19%V in the as-cast condition, showed greater dynamic fracture toughness when compared to the basic Fe-C-Cr alloy, Fig. 8. Fracture toughness was determined mainly by the energy that had to be consumed in extending the crack through ligaments of matrix [7,26]. Since the austenite in that alloy contained very fine  $M_{23}C_6$  carbide particles, higher fracture toughness was attributed to a strengthening of the austenite during fracture. Hence, the contribution to improving the fracture toughness of the alloy containing 1.19 wt.% V, due to the presence of fine  $M_{23}C_6$  carbides within the austenite, was considerably higher than the reduction. Reduction was caused by, on the one hand, increasing the amount of  $M_7C_3$  carbides and reducing inter-carbide distance, and on the other hand, reducing the volume fraction of retained austenite. Where the content of vanadium exceeded 1.19%, fracture toughness decreased (Fig. 8), since the matrix microstructure of those alloys was mainly martensitic.

In heat treated Fe-C-Cr-V alloys with varying contents of vanadium, lower  $K_{id}$  values wear obtained, compared with as-cast alloys (Fig. 8). Several processes which occurred during tempering affected fracture toughness. When held at 500°C for 4h, extensive carbide precipitation probably occurred in the martensite present in the as-cast structure, and retained austenite was decomposed [7,21]. Both effects combined to produce a significant drop in fracture toughness. Furthermore, during the cooling process, after it had been held at the tempering temperature, martensite formed, which also contributed to lower  $K_{id}$  values.

## Conclusions

Vanadium changed the microstructure parameters of phase present in the structure of these alloys, including volume fraction, size and morphology. The degree of martensitic transformation also depended on the content of vanadium in the alloy.

Experimental results indicate that the volume fraction of the carbide phase, carbide size and distribution had an important influence on the wear resistance of white cast iron alloys under low-stress abrasion conditions.

However, the results of fracture toughness tests in both as-cast and heat treated conditions show that the dynamic fracture toughness in white cast irons is determined mainly by the properties of the matrix. Experimental results indicate that the austenite is more effective in this respect than martensite.

The high chromium white iron containing 1.19 wt.% V in the as-cast condition, showed the greater dynamic fracture toughness when compared to other experimental alloys. The higher fracture toughness was attributed to strengthening during fracture, since very fine secondary carbide particles were present mainly in an austenitic matrix.

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