



## MANUFACTURING AND ABRASIVE WEAR ANALYSIS OF MARTENSITIC STEELS REINFORCED WITH ZIRCONIUM OXIDE PARTICLES

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### Abstract

In mining industry and metallurgy, several wearing parts are subjected to the combined effect of abrasive wear and mechanical shocks that requires materials characterized by high hardness in surface and a better toughness in volume. In this present study, martensitic steel which is commonly used in industrial wearing parts is reinforced with zirconium oxides particles as ceramics inserts. Pellets of zirconium oxides that make up the ceramic inserts were prepared and, after casting of martensitic steel, samples were taken from the manufactured parts and characterized. Using ASTM G65 abrasive wear test apparatus, abrasive wear tests of the manufactured steel and, for comparison with other materials most currently used in mechanical industry, were conducted and wear surfaces were also characterized. It was shown that the manufactured steel with zirconium oxides allows improving wear resistance by approximately 44 %.

**Keyword:** Abrasive wear; Ceramics inserts; Manufacturing; Wear resistance; Zirconium Oxides.

### 1.Introduction

In many important engineering fields, wear resistant materials are desiderated. Among the often used materials of wearing parts when wear abrasive resistance and shocks resistances are desired we find chromium cast irons, Hadfield steels and martensitic steels. The famous Hadfield steel has been extensively used for over 120 years because of its excellent wear resistance. This material owns high toughness and mainly excellent work hardening capacity [1, 4]. However, the Hadfield steel has

relatively low strength and hardness properties, and it usually suffers high weight loss and shows short service life under impact-abrasive wear such as rock-crushers. For chromium cast iron, their microstructure were characterized by many high hardness carbides such as M<sub>7</sub>C<sub>3</sub>, M<sub>23</sub>C<sub>6</sub>, M<sub>6</sub>C, M<sub>2</sub>C and MC distributed in matrix composed usually of martensite and austenite with high strength and toughness, so they have excellent wear resistance under extreme wear conditions [5].

The high chromium cast iron or high manganese steel is often used to manufacture the wear-resistance parts. However, the serving life is not satisfactory because it is difficult for the two kinds of materials to resist severe abrasive wear in the process of service. For martensitic steels class, they are commonly used for applications where high mechanical performance is required [6, 8]. They are used as bulk material in a variety of industrial applications, such as hot working dies and tools, propellers, pump impellers, ball bearings and races, bushings, valve seats, industrial knives, etc., where high strength or wear resistance are needed [9]. By opting for the one or other one of these materials most often used in the wearing parts, their life remains however unsatisfactory for a better

production. Since a few years, ceramics particulate-reinforced iron matrix composites are widely used in fields where high hardness, good strength, high wear resistance and temperature resistance are required [10, 12]. Several combinations between particles and various metallic matrices were proposed. The materials have a high hardness and likely to be used as inserts were classified as shown in the table 1 below [13, 15]. Among the various types of particles-reinforced iron matrix such as titanium carbides, tungsten carbides, silicon carbides and vanadium carbides, thorough research were directed towards the development of zircon particles-reinforced iron matrix.

**Table 1.** Mechanical and physical properties of Hard materials.

Material type	Material	Density	Melting point (°C)	Hardness (HV)	Young Modulus (GPa)	Electrical resistivity ( $\mu\Omega.m$ )	Thermal expansion (CDT) $10^{-6}K^{-1}$
Metallic	TiB <sub>2</sub>	4.5	3225	3000	560	7.0	7.8
	TiC	4.93	6.67	2800	470	50	8.0-8.5
	TiN	5.4	2950	2300	250*	25	9.4
	ZrB <sub>2</sub>	6.11	3245	2300	540	6	5.9
	ZrC	6.63	3445	2560	400	42	7.0-7.4
	ZrN	7.32	2982	1600	510	21	7.20
	VB <sub>2</sub>	5.05	2747	2150	510	13	7.6
	VC	5.41	2648	2900	430	59	7.3
	VN	6.11	2177	1560	460	85	9.2
	CrB <sub>2</sub>	5.58	2188	2250	540	18	10.5
	CrN	6.12	1050	1100	400	640	2.3
	WC	15.72	1776	2350	720	17	3.8-3.9
	Covalent	B4C	2.52	2450	3-4000	441	5 10 <sup>5</sup>
BN		3.48	2730	-5000	660	10 <sup>18</sup>	Xxx
C(Diamant)		3.52	3800	-8000	910	10 <sup>20</sup>	1.0
SiC-		3.22	2500	2600	400	8000	4.5
$\beta$ (1000°C)		3.19	1900	1720	210	10 <sup>18</sup>	2.5
Si3N4		3.26	2250	1230	350	10 <sup>15</sup>	5.7
Ionic	AlN						
	Al <sub>2</sub> O <sub>3</sub>	3.98	2047	2100	400	10 <sup>20</sup>	8.4
	TiO <sub>2</sub>	4.25	1867	1100	205	xxx	9.0
	ZrO <sub>2</sub>	5.76	2677	1200	190	10 <sup>16</sup>	11(7.6)
	HfO <sub>2</sub>	10.2	2900	780	Xxx	xxx	6.5
Substract	MgO	3.77	2827	750	320	10 <sup>12</sup>	13.0
	W	19.3	3410	Xxx	407	30	4.45
	WC-6Co	Xxx	Xxx	1500	640	Xxx	5.40
	Acier rapide HSS	xxx	xxx	800-1000	ρ50	xxx	12-15

During manufacturing process of these composites, the main problems which limit their use concern the wettability phenomenon between matrix and the reinforcements which does not allows to have a good adhesion of particles with metal matrix and generate consequently micro-cracks of wear parts during service. According to recent researches, metal matrix reinforced by zirconium oxides are preferred to

others ceramics inserts because it presents a thermal dilation coefficient relatively high 11 10<sup>-6</sup> K<sup>-1</sup> which is very close to the steel coefficient (See table 1). It presents also an excellent abrasion wear resistance and high hardness which is about 1200HV. In this paper, martensitic steels reinforced with zirconium oxides particles was manufactured and characterized.

In order to successfully manufacture the samples by infiltration of the metal in the zirconium preforms which constitutes the novelty of this research, the appropriate casting temperature and metallostatic pressure have been well chosen during casting. Using ASTM G65 abrasive wear test, comparison of the manufactured samples with others conventional steels were also done. Before and after abrasive wear tests, characterizations of all samples were made using conventional methods such as macroscopic observations under low magnifications, metallographic examination, roughness tests and hardness tests.

**2. Materials and methods**

**2.1. Materials**

In this present study, 6 samples of materials were chosen for characterization tests as shown in table 2.

The samples were selected like a reference of the materials commonly used in mining metallurgy and crushing fields. The material A is the manufactured composite with ZrO<sub>2</sub> particles as inserts in the martensitic steel matrix C. The samples D and E are the austenitic steel containing 16.67%Mn and 12.47%Mn respectively, while the sample F is a high chromium cast iron containing 30%Cr.

For indication, the martensitic start temperature (MS) can be determined according C. CAPDEVILA and al. [16] research by the follow equation:

$$M_s(°C) = 539 - 423w_C - 30.4w_{Mn} - 17.7w_{Ni} - 12.1w_{Cr} - 7.5w_{Mo}$$

Where w<sub>i</sub> represent the weight mass of the element i. The MS temperature for all samples is then given on table 3:

**Table 2.** Chemical composition of the materials chosen for tests

Sample/Element	C	Cr	Ni	Mo	Mn
A	0.260	0.148	0.490	0.470	0.600
B	0.615	6.490	0.213	0.267	0.770
C	0.224	0.144	0.500	0.477	0.600
D	1.240	1.250	0.160	0.026	16.670
E	1.040	1.610	0.172	0.024	12.470
F	1.820	30.080	0.120	0.080	1.080

**Table 3.** Martensitic start temperature for samples

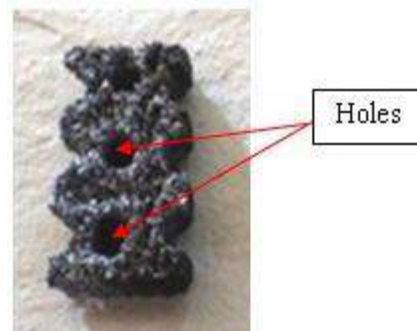
Sample	Ms (°C)
A	397
B	171
C	412
D	-510
E	-303
F	-630

**2.2. Manufacturing martensitic steels metal matrix reinforced with zirconium oxides**

To fabricate samples of martensitic steel reinforced with zirconium oxides by infiltration method (Sample A), pellets were firstly prepared using ZrO<sub>2</sub> particles as shown in figure 1. Using metallic glue as a lubricant, rectangular pellets were compacted and fixed in the sand mold prepared for casting martensitic steel material. As shown in figure 2, the holes introduced in the pellets are conceived to facilitate molten metal infiltration in the performed during casting.



**Fig. 1.** ZrO<sub>2</sub> particles



**Fig. 2.** Example of the compacted pellet

Molten metal was firstly prepared in an induction furnace in which the charge is composed by steel scrap and Ferro-alloys. Before final casting in the sand mold, metal is poured in a siliceous sand mold at a temperature of 1600°C. After remolding the mold, samples have been taken and their macrostructures were examined under low magnification. An example of the manufactured sample with ZrO<sub>2</sub> inserts is shown on the figure 3.

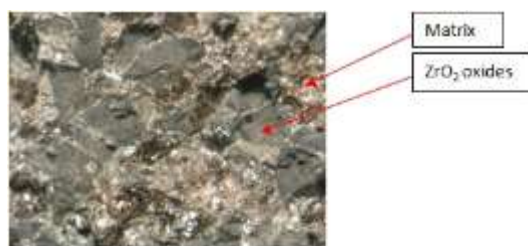


Fig. 3. Example of the manufactured steel

### 2.3. Metallographic characterization and Hardness

In order to examine the microstructures of the materials, cross section of samples were prepared. After conventional and electrolytic polishing techniques using perchloric acid as an electrolyte, the samples were etched with 3% Nital (3ml HNO<sub>3</sub>+98ml ethyl alcohol). Using an optical microscope, microstructures of all samples were examined and characterized. The Brinell Hardness HB of samples was also measured according to ASTM E10 which is a standard method of metallic materials. For each sample, an average of five hardness measurements was taken.

### 2.4. Abrasive wear tests

Using the standard ASTM G65 abrasive wear test apparatus as shown in figure 4, and in order to compare abrasive wear of all samples, abrasive tests were conducted. In abrasive tests, the dry sand/rubber wheel abrasion involves the abrading of a standard test sample with a grit of controlled size and composition. The abrasive is introduced between the sample and a rotating wheel composed with a chlorobutyl rubber tire of a specified hardness. The test sample is pressed against the rotating wheel at a specified force by means of a lever arm while a controlled flow of grit abrades the test surface. The rotation of the wheel is such that its contact face moves in the direction of the sand flow. The abrasive wear test was performed using extra siliceous sand granulometry AFS 45 with a content of silica

particles greater than 99.6%. For each selected material, 3 samples were prepared and tested. The Specimens are weighed before and after the test and the mean mass loss is recorded. After the abrasive wear tests, surfaces of samples were examined and their roughness was also measured.

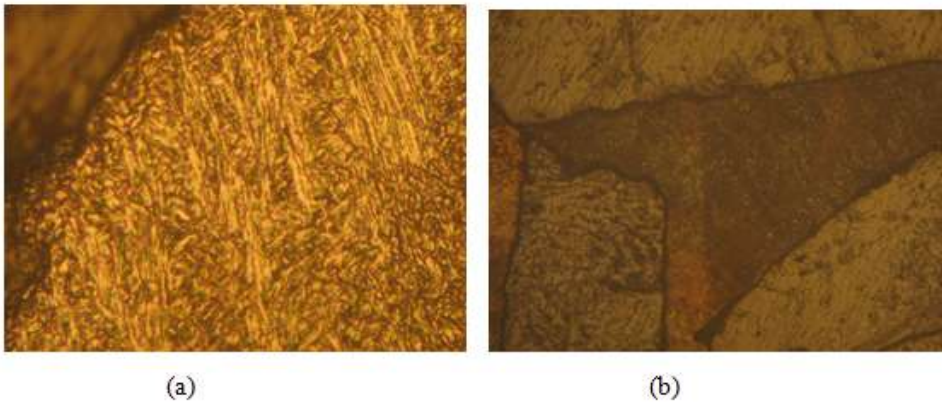


Fig. 4. Abrasive wear test apparatus

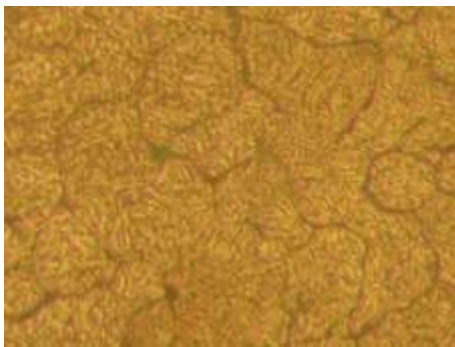
## 3. Results and Discussion

### 3.1. Microstructures and hardness of samples

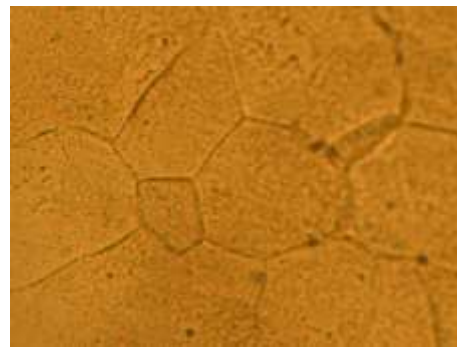
For all samples, the microstructures were firstly examined as shown in the figures 5 to 10. Examination the manufactured sample A (See figure 5) reinforced by ZrO<sub>2</sub> oxides particles as ceramic insert shows a very good adhesion between inserts and the martensitic matrix after casting. For the other materials, their microstructures confirm well the nature of the chosen materials. Indeed, for samples B and C, metallographic analysis reveals martensitic structures which differ only in their morphology. Sample B (figure 6) which contains chromium as an alpha-genic element with a relatively higher carbon content than sample C (figure 7), is characterized by a finer martensitic structure, which is embedded in highly visible grains. For sample B, the observed martensitic structure is coarser. Concerning the samples D and E (figure 8 and 9), the microstructures obtained are austenitic and differ only in their grains sizes which highlight the effect of the manganese like gamma-genic element which stabilizes more the austenitic structure. The micrographs show clearly the manganese effect on the austenitic grains size. Indeed, the higher the manganese content, the grain size becomes larger as shown in the figures 8 and 9. For sample F (Figure 10), the microstructure reveals the presence of primary and secondary carbides in an austenitic matrix related to the high chromium content for this steel.



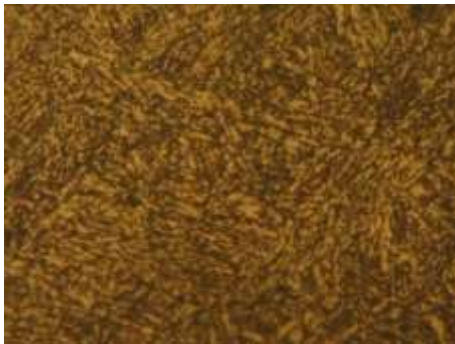
**Fig. 5.** Sample A showing martensitic matrix and ZrO<sub>2</sub> particle under a magnification of 30× (a) and 240× (b)



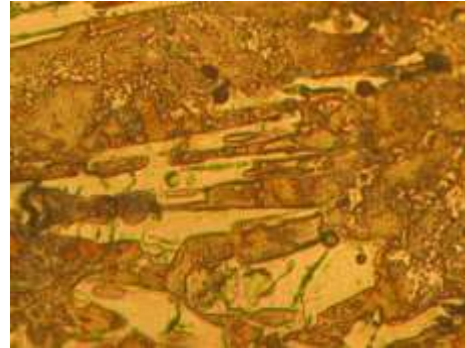
**Fig. 6.** Microstructure of sample B under a magnification of 240×



**Fig. 9.** Microstructure of sample E under a magnification of 240×



**Fig. 7.** Microstructure of sample C under a magnification of 240×



**Fig. 10.** Microstructure of sample F under a magnification of 240×



**Fig. 8.** Microstructure of sample D under a magnification of 240×

For each tested sample, the measured hardness is shown in figure 11 where it can be seen that the manufactured sample A is the one that gives the highest hardness induced by the high hardness of the zirconium oxide particles. The lowest hardness is obtained for the austenitic steel (Sample E) characterized by a low carbon content, comparatively to the sample D although its manganese content is higher. Also, it should be noted that before abrasive wear hardening of the samples D and E, their hardness remain relatively low due to the ductility of the austenitic phase compared to the other tested samples.

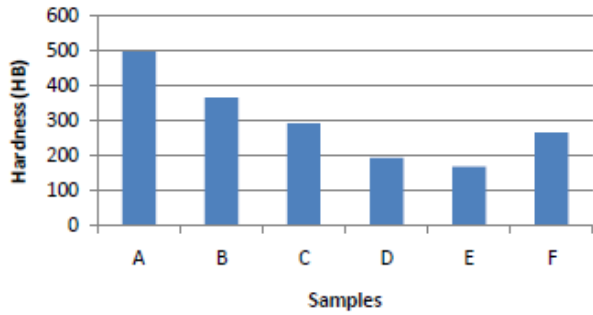


Fig. 11. Mean hardness of samples

**3.2. Abrasive wear tests**

For all samples, mean relative mass loss obtained during abrasive wear tests were shown in figure 12. Examination of relative's mass loss of samples confirm firstly that the martensitic steels metal matrix reinforced with Zirconium oxides is one that have a better wear resistance in comparison to monometallic materials. As the matrix of samples (A) and (C) are the same, it was shown clearly that after abrasive wear test, wear resistance of sample A containing ZrO<sub>2</sub> inserts was improved by 44% approximately. This phenomenon is explained by the presence of ZrO<sub>2</sub> oxides which is characterized by high hardness of about 1200HV.

Mass loss comparison the other samples shows that the sample E is the one which has better resistance to abrasive wear. This is mainly explained by the combined effect of the small grain size observed for this steel, compared to the sample D, and the work hardenability of the austenitic phase. In addition, we

note that during abrasive wear tests, weight loss of samples B, D and F is higher than martensitic steel sample A reinforced with zirconium oxides and steel E.

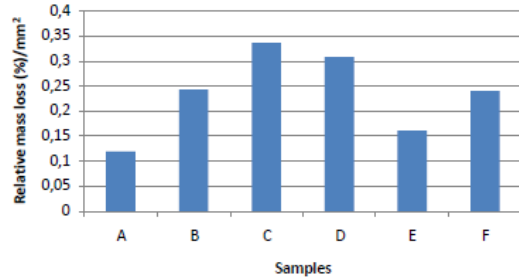


Fig. 12. Mean relative mass loss of the tested samples.

**3.4. Wear surfaces examination:**

After abrasive wear tests, surfaces of samples were examined using a stereo-microscope under low magnification. Morphological aspects of samples surfaces are given in figure 13. For the manufactured steel (Sample A), it was shown that zirconium oxide particles remain fixed and adherent to metal matrix and not dissociate during abrasive tests which proves a good wettability during casting. For other samples, the surfaces are characterized by a uniform removal of the material except for the sample F characterized by a heterogeneity due to the presence of hard carbides in the matrix.

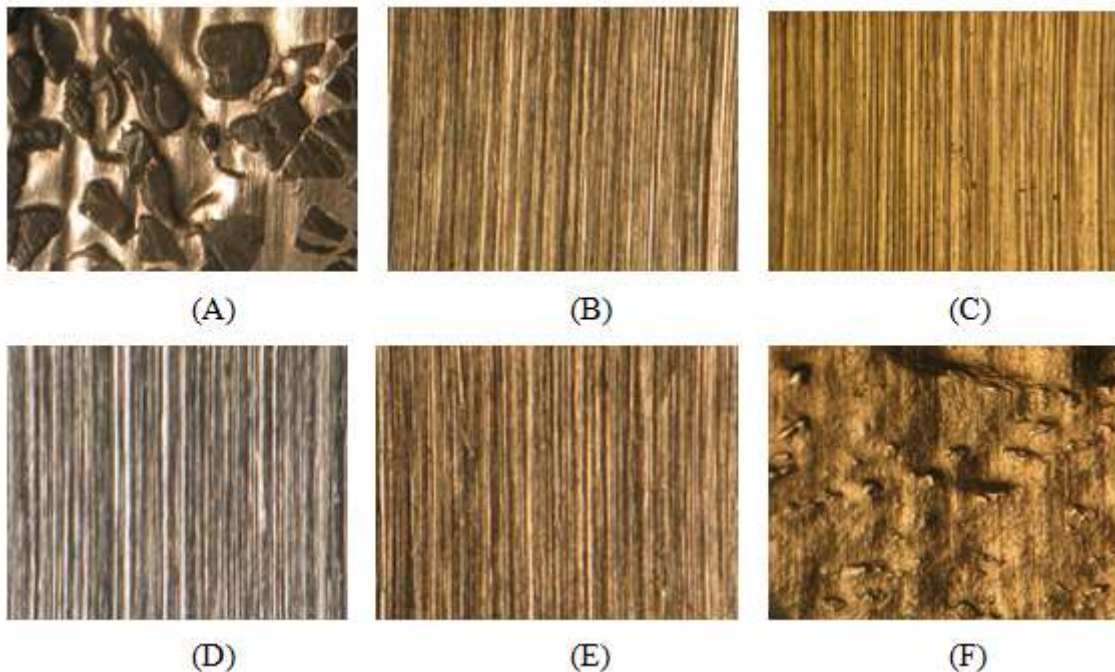


Fig. 13. Morphological aspect of the samples surfaces after abrasive wear tests.

After abrasive wear tests, mean Arithmetic Roughness (Ra) of wear surfaces for all samples are determined and the results are reported in table 4. The highest Ra value is obtained for the sample A which is attributed to the zirconium oxides particles as shown in table 4. For samples B, C, D and E, surfaces roughness was characterized by a small Arithmetic Roughness which varies between 0.68 to 1.23  $\mu\text{m}$ . The sample F which is characterized by an amount of several chromium carbides, the Arithmetic Roughness reaches a value of 1.85. Under the same abrasive wear tests conditions, the high values of the roughness Ra obtained confirm clearly that the surface mass loss is not homogeneous in the presence of hardening particles (case of samples A and F) which improves abrasive wear resistance compared with samples having a low roughness after wear test where the mass loss is homogeneous over the entire surface (Case of samples B, C and D).

Table 4: Ra means values of surfaces of samples.

Sample	Ra ( $\mu\text{m}$ )
A	7.25
B	1.23
C	1.17
D	0.96
E	0.68
F	1.85

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## 4. Conclusion

The zirconium oxides  $\text{ZrO}_2$  as a reinforcement element for martensitic steel allows wearing parts good wear resistance. In comparison with conventional materials commonly used for wearing parts when abrasive wear resistance is desired, the abrasive wear test of the manufactured material shows an increasing of wear resistance of approximately 44%. Also, macroscopic examination, roughness tests and metallographic characterization tests confirm that the presence of abrasive particles, whether they are inserted into the matrix or obtained by metallurgical process during manufacturing, improves the abrasive wear resistance.

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