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INVESTIGATION ON THE INFLUENCE OF TEMPERING ON MICROSTRUCTURE AND WEAR PROPERTIES OF HIGH ALLOY CHROMIUM CAST IRON

ABSTRACT

Mechanical properties, wear resistance and impact resistance of a high-alloy chromium cast iron used in the fabrication of grinding balls have been studied. A rank of tempering heat treatments under several temperatures 500°C, 525°C, 550°C and 575°C was performed after austenitized at 1050°C. The Scanning Electron Microscope (SEM) and X-ray Diffraction (XRD) techniques have been used to characterize the microstructures and identify the phases. The wear balls tests were conducted in a rotating drum with a velocity 0.5 r/s. The tribological tests were carried out by evaluated a weight loss as function time. The measurement of the rebound resilience was determined by Charpy impact tests. The results of XRD showed the presence of the martensite, carbides type M_7C_3 and M_2C for all tempering heat treated. The hardness of the sample increased after the tempering and reach nearly 65 HRC at 1050°C. In another hand, it decreased after the tempering treatment it could be explained by precipitation of the carbides type M_2C .

Keywords: *High-alloy chromium cast iron; microstructure; wear, resilience*

INTRODUCTION

The laboratories has been involved in the production and characterization of metastable and innovative materials. Researcher reach to use efficient of properties of materials alloying in order to increase the best way to refine-grained. The aim of project concerns ball milling which is mainly used in different hard manufactories. The most important field of research, it hope to reach mechanical properties of ball mill. In ball mills, the energy input to the powder charge is came from movement of the cylinder room, In the rotating mills, the steel balls roll along a circular arc on the wall of chamber, and the powder charge is spread on the inner surface of the chamber. Ball mills used in the cement industry used to fragment suspended

particles by stirring a grinding area. They are used to transform the fine rock whose gauge is less than one millimeter (average dimensions are 15 microns) [1-4]. The multiple impacts may damage the ball and reduce the efficiency of the grinding. The mechanical stresses are incurred of diverse origins: shearing due to contact with the wall of the mill, contact between the balls, peeling caused by wear and shock [5-10].

The during life of these materials can be limited by the ability of the grinding ball to resist of periodical mechanical shocks and also depends on its ability to withstand the action of abrasive products with a less mass losses. To link between mechanical and technological requirements, these balls are made of cast high-alloy chromium (10 to 13% chromium) [11-12]. These casts irons are listed in ISO 21988 [13-17]. However, the balls shall be subjected to appropriate heat treatments for improving their mechanical properties. Under these conditions, among the many parameters and criteria to be retained during these heat treatments are the austenitization temperature, the time and tempering [18-22].

For high chromium cast iron with a Cr rate between 11-18 percent (hypo-eutectic composition), the behaviour of solidification begin with the nucleation of dendritic austenite primary (γ), followed by the formation carbides of $\gamma + M_7C_3$ eutectic. The growth mechanism of M_7C_3 carbides and the microstructure formation have been well detail by several researchers [23, 26].

In the previous study of the effect of various kinds of recovering on microstructure and mechanical properties of the high chromium cast iron was studied between 200, 400 and 600 °C. The results showed that, modification of the carbide morphology in cast iron has been greatly improved especially between 450 and 600°C [22]; for this reason we would to investigate on efficient temperature to describe the phenomena of carbide precipitation

The aim of this work is to study the effect of tempering on the microstructure and mechanical behavior of the of high alloy Chromium Cast Iron, we will present the microstructural and structural characterization of the samples at various temperatures by using the scanning electron microscope (SEM), and XRD diffraction. Then the Hardness, wear and resilience had been investigated according to various applied parameter.

EXPERIMENTAL

Materials

The materials used in the present investigation was an high alloy chromium cast iron steel with the following nominal chemical composition (wt %) obtained by using analysis X-rays fluorescence which's represented in table 1.

Table 1. Chemical composition of the high chromium cast iron, wt. %

C	Cr	Cu	Ni	Mn	Fe	Mo	Si	V	Mn
3.0	9-10	0.119	3.0	0.231	Bal	0.2	0.7	<0.01	0.3

It was machined to the cylindrical geometry with 8 mm in diameter. These samples were carried out to hardening heat treatment by an austenitization temperature at 1050°C and tempering at different temperatures from 500°C to 575°C with a step of 25 °C for 5 hours.

Microstructure Investigations

The surface of samples was ground using SiC paper with grit sizes and finally polished with alumina. Microstructures firstly were observed on its surfaces using scanning electron microscope (SEM) in high vacuum, whereas the different phases which is present on samples at different process of heat treatment was investigated using X-ray diffraction, these X-rays diffraction patterns were recorded with a Cu-anode X-ray tube and a curved graphite monochromatic in the diffracted beam.

The presence of blemish in these materials creates a significant background; to improve the ratio of peak/background ratio, an acquisition time of 40 s was used step of 0.04° over the 30° - 100° . The identification of the crystal phases present in the coatings was performed using X'Pert HighScore software supported with the ICDD-PDF2 database.

Mechanical Properties

Rockwell hardness indentation was carried out on the top of samples with a indenter applying a load of 30N. The equipment used is a Wolper kind DIA-TESTOR 2RC hardness meter, the hardness value was took as the mean of 10 measurements.

The wear rate was estimated by the mass losses of thermally treated ball after spending a specified time inside a drum of 98 cm in diameter and 148 cm high. The drum is filled with balls up to 30% of its capacity and driven at 28 cycle/min. The mass was mesured after 15 minutes by weighing using an electronic scale with a 0.1g precision.

The measurement of the rebound energy (resilience test) were performed according to EN 10045 at impact velocity 5.52 m/s. The test method get to the impacting a test pieces with a falling mass, these ball is free to rebound and re-contact after impact druming. There are two methods specified for determining the rebound resilience: the pendulum method as used to measure the energy required breaking once a pre-notched. The equipment used in a pendulum machine is equipped with a clamping device, a centring reference of test specimens and a meter on which you can read the energy after broke. The samples of high alloy chromium cast iron balls have a normalized dimension , the angle through which the samples moves before impact is set to V-notch 45° . The impact resistance KCV is equal to W/S [J/cm^2].

P: Load (N)

h: height (m)

KCV: impact resistance (J/cm^2).

RESULTS AND DISCUSSION

Microstructure analysis of treated ball

The SEM micrograph of the treated ball at $1050^\circ C$ and maintained for 5 hours is shown in Figure 1.

The SEM micrograph showed that the microstructure of almost all treated samples present a significative differences between the intercellular regions and the areas around graphite nodules, as can be seen in Figure 1. Around the nodules, it was observed the presence of bainitic ferrite + retained austenite. Secondly, the intergranular surfaces was composed predominantly of high carbon martensite, probably formed by the transformation of unstable austenite during final cooling. These heterogeneities points out a pronounced segregation of elements during solidification.

Fig. 1. SEM (Backscattered Electron) image of the treated ball held at 1050°C for 5 hours

Chemical elements such as chromium commonly segregate towards eutectic cells boundaries. When we comparing areas around the nodules and intercellular areas, it is like two different alloys with different compositions coexist. This phenomenon allowed to find precipitation of carbides especially area which had high ratio carbon like austenite, whereas in the intercellular areas, it will be transformed to martensite during final cooling. The XRD pattern of the sample treated at 1050°C held for 5 hours is represented by Figure 2.

Fig. 2. X-ray diffraction spectrum of the sample treated at 1050°C held for 5 hours

The X-ray diffraction of our materials show peak of M_2C (ICDD 00-36-1482) carbides which is formed from the precipitation of M_7C_3 (ICDD 00-35-0783) ones, we note also the presence of matrix which is mainly constituted by martensite and austenite phases. Furthermore, a deeply exploration of the Xray diffraction will be investigate by SEM on the same microstructure obtained of the sample treated at 1050°C and hold for 5 hours after that we find a better image showed the presence of carbides type M_2C in the form of small grains type $(Cr, Fe)_2C$ (ICDD n° 017-0333, 031-0619) (Figure 3). The presence of these carbides with differents form and different size is probably due to the holding time which impact on the morphology and the size of the secondary carbides, this form is very beneficial with regard to properties of the matrix [27-28].

Fig. 3. SEM (Backscattered Electron) micrograph of the sample treated at 1050°C held for 5 hours

In order to obtain the efficient mechanical properties and reduce the residual stresses occurred during quenching, the treatment of tempering has been carried out at temperatures between 500°C and 575°C with steps of 25°C and a 5 hours hold each time for all samples.

SEM Image of treated and tempered samples

Microstructures of the treated samples were obtained after four recovery treatments and are shown in Figure 4.

Fig. 4. SEM (Backscattered Electron) image of samples quenched at 1050 °C held for 5 h, a) tempering at 500°C, b) tempering at 525°C, c) tempering at 550°C, d) tempering at 575°C

From the Figure 4 we could note two distinct phases a dark gray phase and another light gray. The microstructure of samples showed a combination of martensite and austenite. The grains had undergone fine recrystallization and these constituted the large part of the microstructure.

At 550°C and 575°C the structure was fully homogenized, during the cooling the microstructure are consisted of fine ferrite grains in which the martensitic (ICDD n° 00-044-1290) was more uniformly distributed. The sample revealed a matrix in which shorter carbides flakes than in annealed sample existed. It was observed that there was many short M_2C flakes surrounded with patches of uniformly distributed matrix grains as seen in Figure 3.

A highly recrystallized matrix grains with some secondary site was observed. This micrograph revealed that the microstructure of tempered samples is presented a number of carbides particles precipitated from the matrix, which indicated that the precipitate carbide particles decomposed by a process of solution in martensitic matrix (JCPDS n° 00-044-1290) [29, 30].

XRD of different samples

Figure 5 showed X-Ray diffraction spectrum of sample had quenching and tempering treatments at 500°C, 525°C, 550°C and 575°C respectively, and they are maintained for 5 hours.

Fig. 5. X-ray diffraction spectrum of the samples treated at 1050°C held for 5 h, a) tempering at 500°C , b) tempering at 525 °C, c) tempering at 550°C, d) tempering at 575°C

Figure 5 shows the diffractogram obtained from the samples treated at 1050°C held for 5 h, and tempering at different temperature, comparison with the ICDD-PDF2 database has allowed the identification of different phases: it's clearly show presence a matrix which is formed mainly by martensite, we note also the formation of residual austenite (ICDD n° 031-0619) which is formed from the treated at 1050 °C. Furthermore, the microstructures did precipitation further growth of chromium carbides within the annealed structure, this carbides consist of Cr₇C₃ (ICDD 00-35-0783) and martensite, M₂C (ICDD 00-36-1482) carbides which form another phase enable this material to improve high surface hardness which promotes resistance to abrasive wear [31]. They are probably due to of decreasing local area of molybdenum, and carbon, in these regions

Mechanical properties

Hardness test

The hardness tests were performed to provide an estimate of the hardness of this new class of material.

Table 2. Hardness of the samples treated at 1050°C and maintained for 5 hours with tempering at 500°C, 525°C, 550°C and 575°C for 5 hours

Heat treatment tempering	Hardness HV	Hardness HRC
Austenitisation at 1050°C/5 h	998	64
Tempering at 500°C/5 h	618	58
Tempering at 525°C/5 h	695	60
Tempering at 550°C/5 h	501	45
Tempering at 575°C/5 h	473	42

The data presented in table 2 show that the hardness measurements were recorded on these same samples. A significant drop in hardness can be noticed as shown in Table 2. The decrease in hardness after the tempering treatment for all treated samples could be explained by the precipitation of M₂C type carbides which results in depletion of the carbon in the solid solution while relaxing residual stresses. Moreover, these M₂C carbides strengthen the surface hardness while maintaining the core of the material malleable thereby promotes resistance to impact. Tempering treatment at 550°C and 575°C, decomposed and broke martensite for produce the ferrite and carbides which decrease hardness of the matrix [32].

Wear resistance

Figure 6 sum up the results of the wear resistance expressed as a relative weight losses in function time of samples treated at 1050°C held for 5 hours and tempering at 500°C, 525 °C, 550°C and 575°C for 5 hours.

Fig. 6. Wear resistance as function time

Cast iron as raw state had a ferrite matrix, using in this state reduced their life time. Their microstructure and mechanical properties can be improved by performing through heat treatments of quenching and tempering.

The important results from this curve are:

- The evolution of this mass losses over time is stayed linear for all 50mm diameter balls treated at 1050 °C held for 5 hours and tempering at 500°C, 525 °C, 550°C and 575°C held for 5 hours.
- The balls tempering at 525 °C, offer the best wear resistance in fact that mass losses noticed are less important. This is explained by the value of the high hardness of the balls
- The grinding balls which have tempering at 500°C, 525 °C, 550°C showed a clear slightly decreasing of mass losses for 30 minutes working and there was a stabilization after this period.
- For the ball tempering at 575°C held for 5 hours, it revealed that the mass loss is increasing regardless in function time but after 60 minutes the mass loss have a significant increase and the acceleration of these mass losses is the highest after 120 minutes. It can be explain by the lower hardness of balls which had an average 42 HRC.

Fracture toughness test

Resilience tests was carried out on different samples treated at 1050°C held after which they have undergone recovery treatments at 500°C, 525 °C, 550°C and 575°C. The obtained values of toughness are shown in Table 3:

Table 3. Average values of the resilience of the samples treated at 1050°C held for 5 hours and tempering at 500°C, 525°C, 550°C and 575°C

Austenisation at 1050°C /5 h and tempering	Resilience K _{CV} (J/cm ²)	Hardness HRC
Tempering at 500°C/5 h	3.75	58
Tempering at 525 °C/5 h	8.75	60
Tempering at 550°C/5 h	5.00	45
Tempering at 575°C/5 h	8.75	42

Through the results obtained from Figure 7, it was found that the samples treated at 1050°C and having undergone recovery treatments at 525°C and 575°C have higher toughness values than those treated and tempering at 500°C and 550°C. This is due to the formation of fine carbides M₂C type in the treated samples at 1050°C; which therefore promotes the impact resistance [33].

Fig. 7. Tempering effect on the impact resistance and hardness

The data depicted in Figure 7 show clearly that the tempering is needed to obtain kind of cast irons with better resilience energy. This behaviour could be explained by the retained austenite which increase the fracture toughness of samples. The higher results of impact energy were 8.75 J/cm² on samples tempering at 550°C; the minus is lead to the samples which is temper at 500°C. In all conditions, it was noted the presence of a process behaviour by the increasing values of impact energy after the increase, which decrease after certain time intervals. For each heat treatment temperature used in this study there is a time interval that produces optimized properties. If the heat treatment time is longer or shorter than the process the mechanical properties will increase.

From the Figure 7 we could say about fluctuation of these results, its nearly linked with microstructure especially the martensitic transformation is about uniform in all the mass. Generally, hardenings followed by a tempering at 525°C and 575°C give better hardness then

that samples tempered at 500°C and 550°C). The quench hardening realized on the grinding balls allows the formation of a hard and resistant martensite matrix (in black on the microstructure in Figure 4). This treatment helps the precipitation of carbides in white on the microstructure (Fig. 4) in the austenite, improves the hardness of austenite due to the reduction of the carbon content and increases the domain of the martensitic transformation. It is always associated with a certain quantity of residual austenite and it has hardness is great. The tempering allows softening the martensite, hence reducing the risks of cracking and reduce impact energy of resilience.

CONCLUSIONS

As result of this work, a choice of an appropriate austenisation temperature as well as an appropriate tempering heat treatment permits improvement of the impact resistance. Consequently, it is recommended: to improve the quality of the cast iron during its elaboration, to prevent infiltration of impurities during casting and to ensure proper distribution of alloying elements.

- There is the presence of M_7C_3 type carbides for all heat treatments carried out.
- Treatments of tempering at temperatures of 500°C and 525 °C provide high hardness. These treatments increase the formation of another type of M_2C carbides is due to the precipitation of M_7C_3 ones.
- The sample which has tempering at 525°C had been the best wear behaviour and impact tensiles. Indeed, in the martensite coming from a recovery heat treatment, fine carbides which perform their function of reinforcing the matrix and the properties of this material are higher than those of the untreated cast-iron state.

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