Effect of heat treatment temperature on the structural evolution of hot forged steel balls used for grinding raw material in cement industry

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Abstract—The raw material preparation sector such as cement works, the steel and mining sector is beginning to pay attention to forged balls as an alternative to the cast balls for their best use characteristics. The forging materials are stronger than the casting material because of their fiber structure. The forging technique, in contrast to the casting process, contributes to a significant improvement of ball wear behavior during the grinding process.

The wear behavior depends on several factors including the chemical composition of the alloy, the type of microstructure and the applied heat treatment. In this work we focus on the effect of austenitizing temperature on the type of the produced microstructure. Al the heat treatment parameters were fixed for all the samples except the austenitizing temperature. Three austenitizing temperatures are selected and a tempering at 250 °C with a holding time of one hour followed by cooling has been carried out. The study is carried out on medium micro-alloyed carbon steel intended for heat treatment.

To make clear the effect of the temperature on the microstructural transformation, several investigations were achieved. Optical and SEM microscopy was carried out to qualitatively characterize the phases A microstructural analysis using the Rietveld method was conducted to access, for each temperature, the type and proportion of phases as well as the crystallites size.

Keywords— Grinding balls, forging, heat treatment, characterization, XRD analysis, Rietveld refinement.

I. INTRODUCTION

The characteristics of materials can be improved by several methods. The addition of alloy elements during the production process [1-4], surface treatments and heat treatments [5] are the most commonly used techniques on a laboratory and industrial scale. The heat treatment is mainly based on the austenitizing operation, the temperature and holding time which are the most important factors in causing homogenization in the austenitic domain [6]. The carbon content defines the austenitizing temperature of the steel to be treated and is closely related to it [7, 8].

Recent work through optimization of heat treatment programs and the chemical composition of steels has succeeded in developing specific microstructures whose matrix consists of bainitic ferrite and a mixture of residual austenite and/or martensite [9-12]. In addition to the nature of the structural components resulting from heat treatment, efforts were made to determine their morphology and distribution in the matrix [13].

H.K.D.H. Bhadeshia [14] showed the importance of adding certain addition elements on bainitic transformation where carbide precipitation can be avoided. The carbides will be replaced, in this case, by thin layers of residual austenite.

The presence of martensite in the microstructure gives a certain range of materials highly desirable properties. Its morphology is determined by the carbon content. In steels with a carbon content of less than 0.6%C the martensite exhibits lath morphology. On the other hand, when the carbon content exceeds 1%, the martensite is in the form of a thin plate [15-20].

Austenitizing allows the chemical homogenization of austenite and also controls its grain size. Temperature and holding time play an important role and take into account the chemical composition of the steel to be treated. Maintaining the austenitizing temperature must be long enough to dissolve the carbides, while avoiding grain grain growing. It ensures dissolution of existing carbides and a homogeneous distribution of alloying elements in order to limit their segregation [21].

From a microstructural point of view, austenite transformation products are characterized by their cell parameters that are a function of the content of insertion elements having low ray atomic, particularly the carbon, which has a notable influence on hardness [22-24].

This work presents the results of the effect of heat treatment temperature on the microstructural transformations of forged steel balls used for grinding raw materials in cement plants. A raw forging sample is chosen as a reference and three austenitizing temperatures were chosen (870°C, 950°C and 1150°C). The aim is to perceive the type of the formed microstructure for each temperature. Optical and SEM microscopies were performed on all the samples. The Rietveld method, used for spectrum refinement, offers us the opportunity to define the proportion of phases, possible changes in the crystalline parameters of each identified phase and the crystallite size.

II. MATERIAL AND METHODS

This steel was supplied in the form of bars with a circular cross-section. It was heated then cut into pieces and shaped by hot forging in suitable dies to obtain a desired final ball shape (Figure 1).

Figure 1: Used steel ball forging die and forged ball

The chemical analysis was performed on a Bruker S8 TIGER X-ray spectrometer on a sample taken from the raw forged steel. Heat-treatment at different temperatures has been achieved after forging. To deduce the effect of heat treatment on the microstructural transformations, three austenitizing temperatures were selected (870°C, 950°C and 1150°C). The holding time was fixed to one hour for all samples. The austenitized samples are quenched in water and subsequently tempered at 250°C with a holding time of one hour and air cooled (Figures 2 and 3).

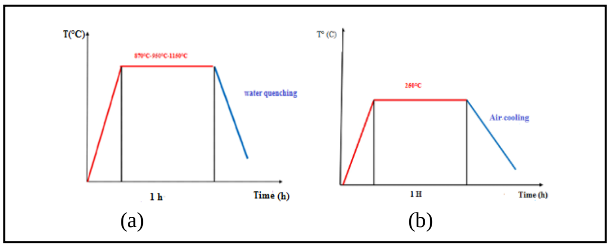

Figure 2: Thermal cycles of quenching (a) and tempering (b)

The X-ray diffraction spectra were recorded using a Brucker D8 advance geometry diffractometer (θ - 2θ) called Bragg-Brentano over an angular range of 0 - 120° in 2θ with a step of 0.02°. The radiation used is copper with a wavelength of λcukα= 1.54056 A°. The adjustment of the diffractograms was carried out using the Rietveld method using the MAUD program (Material Analysis Using Diffraction) [25, 26].
For microstructure characterization, samples are prepared using the conventional metallographic procedure. The optical and SEM microscopies were performed on a NIKON LV150N optical microscope and a EVO/MA25 ZEISS scanning electron microscope respectively.

III. RESULTS

The chemical analysis of the studied material is indicated in Table 1. It is a micro-alloyed steel intended for heat treatment and forging.

Table N°1: CHEMICAL COMPOSITION OF THE STUDIED MATERIAL.

ELEMENTS	***C***	***Mn***	***Cr***	***Si***	***P***	***S***
	0.447	0.59	0.159	0.32	0.003	0.031

The optical and SEM micrographs, in the raw forging state and after heat treatment, are shown in Figure 3. Optical micrographs in the raw forged state reveal the presence of a light-colored network whose orientation varies from one area to another. This network can be a bainitic or troostic ferrite which is in favor of a fine mixed structure.
The forging process requires heating the material to about 1150°C, then shaping it and finally cooling it in water. During this operation, the forged material undergoes a phase transformation similar to that of a hardening treatment. Hot forging reduces microscopic voids between massive crystals formed during primary metal solidification and it also decreases certain surface defects [27]. It has also been reported that the carbon content has a remarkable influence on the morphology of martensite. This latter is lath shaped in heat treated steels with carbon content less than 0.6% C [28, 29].
The solidification of this steel takes place according to the Fe-C diagram; its microstructure in its as cast state is ferrito perlitic.

In its raw forged state, the studied steel reveals a microstructure whose phases follow a well-defined direction, illustrating a stacking that confirms the forging process that was undergone by the sample. In this state, the microstructural component appears as a lengthened morphology oriented in a defined direction. The micrographs show the structural differences between the forged steel samples after the different heat treatment temperatures. It can be seen that the as forged microstructure is finer than that of the heat treated steel at 870°C and 950°C. The microstructures after treatment at 870°C and 950°C are similar in terms of phase type but not of phase proportion. At 870°C, a dominance of clear area is clearly noticed. After austenitizing at 1150°C and tempering at 250°C, the chemical etching highlighted some phases which are clearly delimited by their color.

The X-ray spectra obtained on the different forged steel samples are shown in fig. 4. The refinement of the XRD spectra recorded on the samples show the type of existent phases (fi.5). For each phase correspond an intensity and a 2theta diffraction angle. This technique shows that the microstructure of the studied material is composed of a mixture of martensite and bainite or troostite. The bainite and troostite are a kind of aggregates of alpha iron and cementite, they are not identified by the XRD technique but their presence is confirmed by the detection of alpha iron phase.

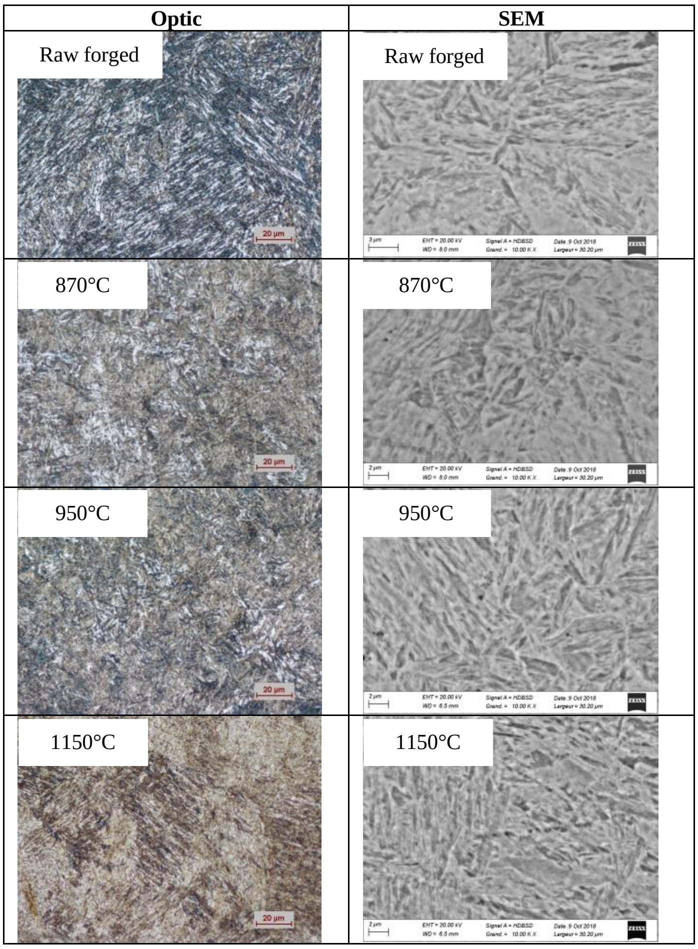

Figure 3: Optical micrographs and SEMs of raw and heat treated forging samples

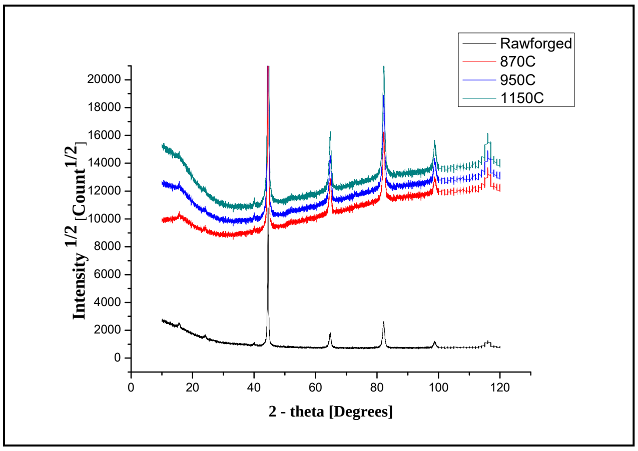

Figure 4: XRD patterns of the studied materials

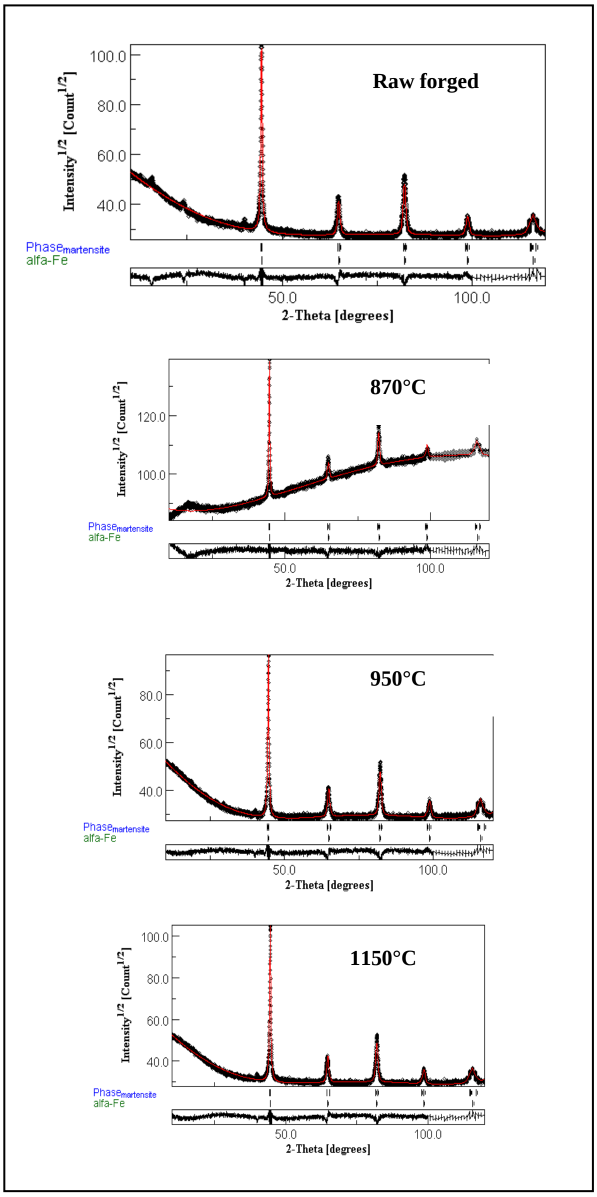

Figure 5: Rietveld refinement of X-ray patterns of the raw forged and heat treated steel balls

The variation in the proportion of phases for the raw forged and heat treated states as a function of the processing temperature is shown in fig.6. The alpha iron phase here referred to represent bainitic ferrite. The studied steel, even in the raw forged state, has a mixed microstructure of martensite and bainite. The quantitative analysis of the various samples shows that the studied steel is predominantly bainitic. The different phases are well shown on the diffractogram. The intense peaks correspond to the most abundant phase. The bainite is revealed here by the bainitic ferrite. This ferrite has been cited by several authors and has been identified with acicular or irregular lattice morphology [30-34].

The variation in the treatment temperature contributed to a variation in the proportion of existing phases (fig. 6). The proportion of phases at 870°C is closer to 50% for each identified phase. As we also note that in the raw forging and in the treated state at 950°C, the quantitative analysis of the samples by XRD gave very close phase proportions.

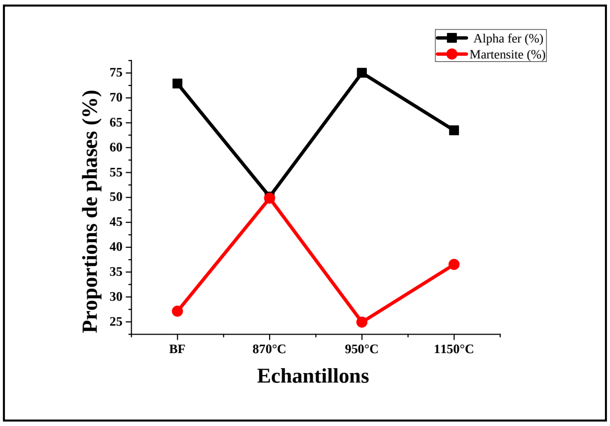

Figure 6: Variation of the proportion of phases as a function of the treatment temperature

The variation in crystalline parameters of the microstructural constituent of steel in the raw forged state and after heat treatment is shown in Figure 7. There is a proportional relationship between the austenitizing temperature and the value of the cell parameter of bainitic ferrite. The variation of the cell parameters can be assigned either to compression or expansion of the crystal lattice. An increase in the parameter of the crystalline mesh of ferrite is observed with the increase of the austenitizing temperature. This explains why the cell parameter bainitic ferrite has expanded after heat treatment.

The variation in processing temperature, compared to the raw forging state, also has an effect on the value of the martensite cell parameter.

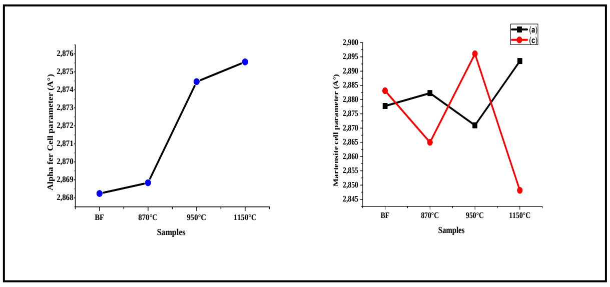

Figure 7: Effect of heat treatment temperature on the cell parameters of ferrite and martensite

Fig. 8 shows the variation in the c/a ratio of martensite. This parameter expresses the compactness of the crystal lattice of the martensite in the raw state of forging and after heat treatment. Fig. 8 shows that the compactness of martensite decreases at 870°C reaches a maximum at 950°C and then decreases to a minimum at 1150°C.

The effect of heat treatment on crystallite size is shown in Figure 9. A slight decrease in bainitic ferrite crystallite size is observed between the forging raw state and after treatment at 870 ° C. A significant increase is noticed at 950°C and 1150°C. The increase in austenitizing temperature has led to an increase of the bainitic ferrite crystallite size. The martensite crystallite size records an increase between the raw forging state and 950°C than drop to a minimal value when the the austenitizing temperature rise to 1150°C.

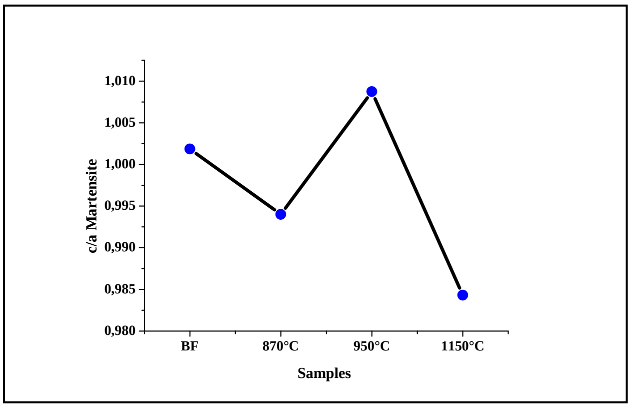

Figure 8: Change in c/a martensite ratio

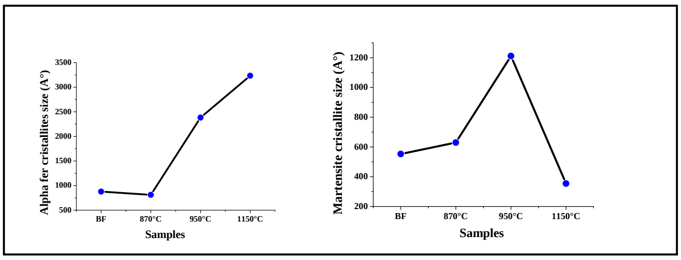

Figure 9: Effect of treatment temperature on crystallite size of microstructural constituents

Conclusion

This study was carried out on a forged steel intended for ball mill grinding. The aim consists to study the effect of the study the effect of the heat treatment temperature on the microstructural changement.

Affecting a change of the austenitization temperature with maintaining constant all the other heat treatment parameters led us to deduce the following conclusions:

- In general, the microstructure of the steel studied, in the raw forged state and after heat treatment, consists of a mixed microstructure constituted of martensite and bainite.
- The Rietveld refinement of the XRD spectra recorded on the different samples has allowed detecting the nature and proportion of the existing phases. The effect of the treatment temperature was also observed on the cell

parameters variation as well as on the crystallites size of the different existing phases.

- The variation in the treatment temperature contributed to a variation in the proportion of existing phases. The proportion of bainitic ferrite is always higher than that of martensite except at 870°C where the proportion of the two phases is approximately identical. A proportional relationship between the austenitizing temperature and the bainitic ferrite cell parameter is observed.
- The quantitative analysis does not reveal the existence of retained austenite nor cementite in all the samples.
- This study requires further analysis using advanced investigation techniques in order to detect and quantify the microstructural characteristics of each identified component.

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