Effect of Austenitizing Temperature on The Structural Evolution of Hot Forged Steel Grinding Balls

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Abstract: This paper reports the influence of heat treatment temperature on the microstructural changes that may occur in an XC38 forged steel grinding ball. Three austenitizing temperatures (870°C, 950°C and 1150°C) were used and a tempering at 250 °C followed by air cooling was carried out. Optical and scanning electron microscopies, as well as X-ray diffraction, were performed to investigate the microstructure and phase of the different samples. Microstructural analysis using the Rietveld method was conducted to access, for each temperature, the type and proportion of phases as well as crystallites size and microstrain. The obtained results were compared to those recorded on as forged steel. The effect brought by the variation of the austenitization temperature was well highlighted by the obtained results. The nature and proportion of microstructural phases were significantly affected by the increase of the austenitization temperature which was in favour of the increase of martensite content in a mixed microstructure consisting of bainite and martensite. A rising amount of martensite is noticed, with the increase of temperature, at the expense of the bainite content. An effect was also noticed on phase’s proportions, microstructural parameters, crystallites size and microstrain.

1. Introduction

Materials characteristics can be improved by several methods. Addition of alloying elements during the production process, surface treatments and heat treatments are the most commonly used techniques [1-5]. Heat treatment is based on austenitizing operation, temperature, holding time and cooling media. These parameters are the mainly important factors causing homogenization in the austenitic domain [6-8]. Recent work through optimization of heat treatment programs and 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, 13]. In addition to the nature of the microstructural components resulting from heat treatment, various attempts have been deployed to assess their morphology and distribution in the matrix [14, 15]. Other research works showed the importance of some alloying elements addition on a bainitic transformation where carbide precipitation can be avoided and replaced by thin layers of residual austenite [16]. The presence of martensite in the microstructure gives a range of materials with high desired properties [17, 18]. 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. However, when the carbon content exceeds 1%, the martensite is of thin plate [19-24].

Austenitizing treatment allows a chemical homogenization of the austenite and 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. The austenitizing holding time must be long enough to dissolve the carbides while avoiding grain growing. It ensures the dissolution of existing carbides and a
homogeneous distribution of alloying elements in order to limit their segregation [25]. From a microstructural point of view, the 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 [26-28].

The aim of the present study is to investigate the influence of different austenitizing temperatures on microstructure and the effect that may occur on the microstructural parameters. The selected heat treatments consist of quenching and tempering. The goal is to perceive, for each heat treatment temperature, the obtained microstructure. Optical and SEM microscopies were performed on all the samples. The Rietveld method, used for XRD spectra refinement, offers the opportunity to define the type and proportion of existing phases, the variation of cell parameters that may occur in each identified phases, the crystallites size and also the microstrain.

2. Materials and method
Bar steel with a circular cross-section is used in this work. It was heated then cut into pieces and shaped by hot forging using suitable dies to obtain a desired final ball shape (figure 1). These balls are intended for grinding materials.

![Manufacturing process of forged balls.](image)

The chemical analysis (table 1) was performed on a Bruker S8 TIGER X-ray spectrometer. Heat-treatment cycles were achieved after forging. A raw forging sample is selected as reference and three others are heat-treated at 870°C, 950°C and 1150°C. The austenitized samples are quenched in water, subsequently tempered to 250°C and the holding time was set at 1 hour for both cycles of heat treatments. Thereafter the samples are air-cooled (figure 2 (a and b)).

![Thermal cycles of quenching (a) and tempering (b).](image)

Samples intended for microstructure characterization are prepared by using the conventional metallographic procedure. The optical and SEM microscopies were performed on a NIKON LV150N optical microscope and an EVO/MA25 ZEISS scanning electron microscope respectively. The X-ray diffraction spectra were recorded on a Brucker D8 advance geometry diffractometer (0-20) called Bragg-Brentano over an angular range of 0 - 120° in 20 with a step of 0.02°. The radiation used is
copper with a wavelength of $\lambda_{\text{CuK}\alpha} = 1.54056$ Å. Phases identification, lattice parameter estimation, crystallite size and microstrains were deduced from the refinement of X-ray patterns using Maud software based on the Rietveld method [29-31].

3. Results

The chemical analysis of the studied steel is listed in Table 1. It is a micro-alloyed steel intended for heat treatment and forging. The steel solidification takes place according to the Fe-C diagram; its microstructure in the as-cast state is made of ferrite and perlite.

| Table 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 forging process requires heating the material to about 1150°C, then shaping it. After forging, the steel is air-cooled, so, it undergoes phase transformation similar to that taking place during hardening treatment. The hot forging process reduces microscopic voids between massive crystals formed during primary metal solidification and it contributes to a decreasing of surface defects [32] and promotes fibrous microstructure.

Heat treatment and cooling media affect the steel microstructure. To meet precise usage requirements, it allows the precipitation of well-defined microstructural phases. Also, it had been reported that the carbon content had a substantial influence on the martensite morphology. It was lath shaped in heat-treated steels with carbon content less than 0.6% C [33, 34]. Figure 3 shows optical and scanning electronic micrographs of raw forged and heat-treated forged grinding balls. Optical micrographs in the raw forged state reveal the presence of a light-coloured network whose orientation varies from one area to another. The microstructural components appear as a lengthened morphology oriented in a defined direction. The micrographs show the structural differences between the forged steel samples after the various heat treatment temperatures. It can be seen that the as-forged microstructure is finer than that of the forged 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. At 870°C, a dominance of clear area is noticed. After austenitizing at 1150°C and tempering at 250°C, the chemical etching highlighted some phases which are clearly highlighted. The studied steel, even in the raw forged state, has a mixed microstructure.

The analysis of diffraction peaks profiles allows characterizing the structural changes induced by austenitizing temperature variation. X-ray diffraction (XRD) patterns recorded for the steel in the raw forged state and after different heat treatments are shown in Figure 4. XRD pattern of the raw forged steel shows the Bragg peaks of bcc-Fe, while peaks of alpha iron and martensite are revealed on the heat-treated steels. Peaks of alpha iron are indexed according to the bcc (space group Im-3m) structure with parameter lattice $a = 0.2869$ nm and peaks of martensite are indexed according to tetragonal (space group I4/mmm) structure with lattice $a = 0.2886$ nm and $c = 0.2862$ nm. Except the raw forging state, all the analyzed samples are composed of ferrite and martensite. No diffraction peaks arising from retained austenite or cementite were detected in the raw forged state or after heat treatment. Austenitizing sample at 870°C contributes to an observable change in microstructural phases. After austenitizing at 870°C, a quadratic martensite Brag pic diffusion appeared and still observed, after 950°C and 1150°C. With varying the austenitization temperature, the same diffraction peaks are detected. The alpha iron is referred to represent the bainitic ferrite. Several authors reported that this ferrite appear with acicular or irregular lattice morphology [35-39]. On the figures 5-8 are reported respectively, the effect of the austenitizing temperature on volumetric fraction, cell parameters, crystallites size and microstrain of the identified microstructural constituents. The MAUD results show that the raw forged steel exhibits a completely bainitic microstructure. As the austenitization temperature increases, the content of bainite decreases to the detriment of martensite to about 50% at 1150°C.

The microstructural parameters, obtained by Rietveld refinement of the X-ray diffraction patterns recorded on the raw forged and heat-treated forged steel are shown in Figure 6 (a, b and c). In Fig 6.a, an increase in the parameter of the ferrite cell parameter is noticed when the austenitizing temperature
rises then dropped (0.28499, 0.28446, 0.28867, 0.28595nm) at 1150°C. The change in processing temperature, compared to the raw forging state, also has affected the value of the phase’s cell parameter.

Figure 3. Optical and SEM micrographs of raw and heat-treated forged steel samples.
Figure 4. Rietveld refinement of X-ray patterns of the raw forged and heat-treated steel samples.
Figure 5. Volume fraction of microstructural phases.

Affecting an increase of the austenitization temperature (figure 6) results in a change of the martensite cell parameters. An inversely proportional relation is observed between “a” and “c” martensite cell parameters (figure 6). The martensite cell parameter “a” is similarly affected by the heat treatment temperature as alpha iron cell parameter. On the other hand, the martensite cell parameter “c”, compared to that obtained at 870°C, decreases at 950°C and rises at 1150°C to reach an intermediate value. The effect of heat treatment on crystallite size is shown in Figure 7. Through the performed heat treatments, a gradual increase of the α-Fe crystallite size is observed.

It has been shown that the variation of the crystalline parameters induces difference of the crystallite size [40-45]. No relationship is perceived between the α-Fe cell parameter and its crystallite size. A slight decrease in bainitic ferrite crystallite size is observed between the raw forging state and after treatment at 870°C, then a substantial increase is noticed at 950°C. The increase of austenitizing temperature has led to an increase in the bainitic ferrite crystallite size. Unlike, the average size of martensite crystallites records a decrease at 950°C and then increases to 1150°C.
Figure 6. Phase’s cell parameter as a function of austenitizing temperature
(a): “a” alpha iron; (b): “a” Martensite; (c): “c” martensite.

Figure 7. Crystallite size versus heat treatment temperature.

Figure 8. Crystallite size versus heat treatment temperature.

The alpha iron and martensite microstrain values in the raw forged steel and after the various heat treatment temperatures are indicated in figure 9 (a, b). An inversely proportional relationship between the average crystallite size and the micro stain of martensite is clearly visible. Nevertheless, this
relationship is not well highlighted by alpha iron. Hence, the austenitization temperature appears to have a weak influence on the relationship between the alpha iron crystallites size and micro stain. As the austenitization temperature increases the microstresses decrease.

Figure 9. Effect of treatment temperature on microstructural constituents micro-strain  
(a): Alpha iron; (b): Martensite.

4. Conclusion
The effect of the austenitizing temperature on the microstructural transformation of hot forged steel has been addressed in this work. The study was carried out on forged grinding media. It was observed that when affecting a change of the austenitization temperature with maintaining constant all the other heat treatment parameters a mixed microstructure made of martensite and bainite expands. XRD technique and Rietveld refinement method have 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 change in the heat treatment temperature contributed to a variation in the proportion of the existing phases. The proportion of bainitic ferrite is always higher than that of martensite except at 1150°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 detected. Although, there is no perceived relationship between the α-Fe cell parameter and its crystallite size. Conversely, a relationship between the average crystallite size and microstructural microstrain stress is perceived. Neither residual austenite nor cementite was detected in the microstructure of all the samples.

Acknowledgements
The authors acknowledge the priceless support of Dr. Samia BOUDEBANE LEMBOUB, Head of Department of Materials Science and Engineering, for carrying out the analyses by scanning electron microscopy.

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