Abstract

Ductile iron, also referred to as nodular cast iron or spheroidal graphite iron, is a type of graphite rich cast iron containing graphite in the form of spherical nodules. The mechanical properties of ductile iron are determined by the size, number fraction and nodularity of graphite particles as well as by the matrix phase(s). Depending on composition and actual cooling rate, the matrix of as cast ductile iron is usually composed of various fractions of ferrite and pearlite. Since the overall mechanical properties are significantly influenced by the matrix phase fractions, determination of pearlite content in as-cast ductile irons is particularly important. Magnetic Barkhausen Noise (MBN) technique provides a good alternative to traditional metallographic examinations in terms of its fastness and non-destructive nature. The present study aims at investigating the possibility of using the MBN-technique for non-destructive determination of pearlite content in ductile iron castings. For that purpose a set of 4 specimens were produced from the same ductile iron casting to ensure that all specimens contain identical size, number fraction of nodularity of graphite particles. The pearlite fraction of the samples were determined by metallographic examination and the MBN-response of the samples were evaluated by the peak position, shape and amplitude of the signals obtained by a commercial MBN-system. The results show that the pearlite fraction in the matrix influences the hardness, all of which also effect the MBN signals. This indicates that MBN-technique can be utilized for evaluating the matrix phase fractions of ductile iron castings.

1. Introduction

Ductile cast iron has been in production since 1950s and nowadays over 25 million metric tons are cast yearly [1]. Heat treatment applications have a strong effect on the properties of cast irons, specifically the ratio of ferrite to pearlite have a pronounced effect on mechanical properties [2, 3]. Since heat treatment is the latest processing step, controlling the formation of required microstructure afterwards is of great importance. Traditionally, X-ray diffraction (XRD), metallographic examination via optical and scanning electron microscopes were the main methods used to control the microstructure. On the other hand, MBN measurement provides a good alternative to aforementioned traditional techniques in terms of its non-destructive nature and fastness. The MBN method is based on ferromagnetism phenomenon. Ferromagnetic materials below their Curie temperature retain a large spontaneous magnetic moment due to the cooperative alignment of unpaired electron spins along a common direction. Oppositely magnetized domains divided by domain walls form to minimize the magnetic energy. The change in magnetization, caused by the application of the external magnetic field, takes place by movement of the boundaries between domains in weak fields or by rotation of the direction of magnetization in strong fields. On removing the field, the magnetization again declines to zero if there is no hindrance to domain wall motion [4-5]. Various studies have been published on characterization of steel microstructures non-destructively. The ferrite-pearlite fraction in cast irons influence had been reported to influence magnetic properties, especially the magnetic relative permeability [6]. Moreover, eddy current method has been used to estimate pearlite fraction [7, 8]. The present work aims at evaluating the microstructure of nodular cast irons non-destructively by MBN.

2. Experimental Procedure
GGG70 nodular cast iron specimens of 20 mm thickness and 20 mm diameter, whose chemical composition is given in Table 1, were used in this study. The heat treatment of the specimens include inter-critical annealing at 6 different temperatures (830 °C, 820 °C, 810 °C, 800 °C, 790 °C, 780 °C) for 30 min., followed by air cooling to room temperature. A specimen was left in as-cast condition.

Table 1. Chemical composition of the GGG70 cast iron (wt%)

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Cr</th>
<th>Mo</th>
<th>Mn</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>3.8</td>
<td>0.02</td>
<td>0.63</td>
<td>0.3</td>
<td>0.02</td>
</tr>
<tr>
<td>Si</td>
<td>2.6</td>
<td>0.045</td>
<td>0.035</td>
<td>0.012</td>
<td>Bal</td>
</tr>
</tbody>
</table>

After the heat treatments, all specimens were prepared for metallographic examination. After standard grinding and polishing, the specimens were etched with 4% picral solution. Then, all specimens’ surface sections were examined by Leica DMI 5000M optical microscope under bright field illumination. For each specimen, 10 micrographs from randomly selected regions were taken at 100x magnification. Those micrographs were then analyzed using Leica Application Suite to determine the volume fraction of ferrite and pearlite. Moreover, an average Brinell hardness value was determined using Emco Duravision 200 instrument, using 2.5 mm indenter-ball under 187.5 kgf load. MBN measurements were performed by using commercial system (Rollscan, μscan 600). The general purpose sensor S1-18-13-01 was used for the MBN measurements. A sinusoidal cyclic magnetic field was induced in a small volume of the specimen via a ferrite core C-coil. The Barkhausen signals were collected at a sampling rate of 2.5 MHz and then filtered with a wide band-pass filter (10-1000 kHz). A total of 10 signal bursts, each of which represent one half of the magnetization cycle, were used to analyze the Barkhausen signal of each specimen. 6V of magnetizing voltage and 250 Hz of magnetizing frequency was used to obtain reliable MBN profiles.

3. Result and Discussion

Optical micrographs of the specimens are given in Figure 1, which show that typical nodular graphite particles on a matrix of ferrite-pearlite mixture had been obtained in the present ductile cast iron specimens. Figure 2 shows the ferrite and pearlite fractions as a function of annealing temperature. As cast specimen has the highest pearlite fraction and the 780°C inter-critical annealed sample has the lowest fraction. As inter-critical annealing temperature increases pearlite fraction increases, and ferrite fraction decreases. The increasing pearlite content increases the hardness as well, which is shown in Figure 3.
Figure 1. Optical micrographs of the specimens inter-critically annealed at the indicated temperatures, taken at 100x magnification under bright field illumination.

Figure 2. Ferrite and pearlite fractions of the specimens as a function of inter-critical annealing temperature.

Figure 3. Brinell hardness values of the specimens.

MBN measurements provide a representative noise signal which is obtained in a small volume of the specimen by applying alternating magnetic field. The irreversible process of magnetization which is related to the dynamic behavior of domains (nucleation, annihilation and growth of domains) affects the detected noise signal. Grain boundaries, dislocations and precipitates, in other words the microstructure of the material directly influences this dynamic behavior.

The raw magnetic noise data consists of series of voltage pulses and associated magnetic field values. A range of different analysis methods and parameters have been used to express the MBN response [9, 10]. In the present study, the strength of the MBN signal is quantified with the root-mean-square (RMS) value which is calculated according to the following formula:

$$RMS = \sqrt{\frac{1}{n} \sum_{i=1}^{n} x_i^2}$$  \hspace{1cm} (1)

Where $x_i$ represents the MBN signal amplitudes that passed through the wide band-pass filter specified in the experimental section.

The microstructural features that impede the dislocation movement, also inhibits the domain wall motion. Therefore an excellent correlation is expected between MBN signals and hardness [10]. Figure 4 shows the correlation between hardness and RMS of MBN of the present specimens. The hardness values shown in Figure 3 were used for the correlations. Those values represent the hardness of the matrix, which is composed of pearlite and ferrite.

Figure 4. Correlation between hardness and RMS.
Figure 5. Correlation between pearlite fraction and RMS

The matrix phases influence the domain wall displacement; therefore, the correlation between the area fraction of pearlite and the RMS response is also studied and the results are given in Figure 5. Those correlations were determined via a simple linear regression using least squares method. The goodness of fit was quantified by the $R^2$ value, and also shown in Figures 4 and 5. The calculated $R^2$ also show that MBN signal depends on the microstructure and is sensitive to microstructural variations.

The specimen that was inter-critically annealed at 820 °C has the lowest RMS value due to the harder matrix, which allow smaller domain wall displacements. Lowering the annealing temperature decreases the hardness of the matrix and enhances the domain wall displacements. Therefore, MBN activity in nodular cast iron increases with decreasing annealing temperature.

It also should be noted that for the present case only the matrix phase fractions and the hardness correlations are considered. However, the size, shape and volume fraction of graphite particles can also influence the MBN response of the specimens. Moreover, the present case concentrates on pearlite fractions between 45 - 55%. Nevertheless, this preliminary study clearly shows the potential of the MBN technique for non-destructive microstructure characterization of nodular cast iron.

4. Conclusions
The evaluation of microstructure by using MBN measurements had been studied on a set of GGG70 ductile cast iron specimens inter-critically annealed at different temperatures. The following conclusions can be drawn:

- Inter-critical annealing at different temperatures followed by air-cooling can vary the volume fraction of ferrite and pearlite in GGG70 nodular cast iron.
- As inter-critical annealing temperature increases the volume fraction of pearlite increases which also increases matrix hardness.
- MBN method is capable of evaluating the influence of inter-critical annealing on the microstructure of ductile cast iron. This technique can be utilized for evaluating the pearlite fraction and controlling the formation of the desired microstructures in ductile cast irons by establishing quantitative relationships between the microstructural variations and MBN response of the materials.

5. References

[1] 50th Census of World Casting Production, Modern Casting, December, 2016