DETECTION OF AUSTENITE TRANSFORMATION OF ADI CAST IRON USING ELECTROMAGNETIC SENSOR

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Abstract
An electromagnetic sensor was used to evaluate the microstructure in austempered ductile cast iron for austempering at varying temperature. Microstructural changes in cast iron as a result of changes in the permeability were observed. The electromagnetic sensor could be used for detection of bainite and martensite at isothermal treatment within specific temperature range. Light optical metallography revealed that samples microstructure contains bainite, martensite and austenite. Based on theoretical assumption, the value of the inductance difference should be directly proportional to volume of austenite phase in microstructure. The device could be used for the investigation of multiphase iron alloys of different ratio microstructure phases.

Key words: bainite, austempered, ductile, iron.

INTRODUCTION

Commercial iron products are produced using heat treatment procedure in which the austenite transformation is cooled continuously at varying temperature resulting into a mixture of different microstructure transformation products namely ferritic or martensite (ZHANG ET AL., 2012; YIN ET AL., 2002; PIETRZYK AND KUZIAK, 2011). However, the different transformation products of the microstructure are based on different mechanism and mechanical properties of the products (GAO ET AL., 2011; CHEN ET AL., 2012). To obtain a particular microstructure, the transformation product of austenite is transformed without rapid cooling at constant temperature or within a temperature limit according to isothermal transformation of austenite cooling kinetics. The monitoring of isothermal transformation of austenite especially in real steel/cast iron is difficult therefore the knowledge of isothermal heat treatment from time temperature transformation diagram is applied (LEE ET AL., 2010). The process of austenite transformation depends on the chemical composition and austenitizing temperature and time respectively.

Electromagnetic sensor (EMS) has been reported to be suitable for the detection of phase transformation in carbon steel cooling below the Curie temperature (T_c) (YIN ET AL., 2007; WANG ET AL., 2014B, 2014A; REISSMAN ET AL., 2012; PACURAR ET AL., 2012; LIU ET AL., 2013; GHANEI ET AL., 2013). The electromagnetic field created by the EMS is sensitive to any variations in the electrical resistivity and magnetic permeability of the sample resulting in changes in the complex trans-impedance value recorded. This is particularly useful because resistivity and permeability are known to be directly influenced by the microstructure and carbon content. Austenite is paramagnetic ($\mu_r = 1$) whereas ferrite, pearlite, bainite and martensite are ferromagnetic below T_c ($\mu_r = 200+$). Hence EMS is able to detect the ferromagnetic phase change below T_c. But microstructure phases in iron products (steels, cast iron) contain ferrite with $T_c \approx 770$°C, cementite Fe_3C with $T_c \approx 210$°C or M_7C_3 carbide with $T_c \approx 250$°C. Magnetic permeability of martensite and cementite phase is less than ferrite phase therefore an increase in the martensite percentage decreases the magnetic permeability of the material leading to a reduction in the impedance. The isothermal heat treatment of austempered ductile iron (ADI) is from 270°C up to 400°C, therefore only ferrite as ferromagnetic material can be detected by electromagnetic sensor during isothermal heat treatment whereas cementite and graphite are paramagnetic at temperature limit.

The mechanical properties such as strength, toughness and wear resistance depend on ratio of microstructure phases (KOLAŘÍKOVÁ ET AL., 2013; CHALA ET AL., 2005; CHOTĚBORSKÝ AND HRABĚ, 2013;

CHOTĚBORSKÝ ET AL., 2009; ZHANG ET AL., 2012). The use of an isothermal heat treatment can determine the mechanical properties of austenite transformation products (HUNG ET AL., 2002; ERFANIAN-NAZIFTOOSI ET AL., 2011; ZHANG ET AL., 2012). The actual ratio of austenite transformation products is usually controlled off-line by metallography after heat treatment. The present study investigates the relationship between electromagnetic sensor (EMS) and signal of a micro-structure austempered ductile iron (ADI cast iron).

MATERIALS AND METHODS

ADI cast iron samples with dimension of 8 mm x 25 mm x 50 mm were used for the isothermal heat treatment. EMS (40 threads and 4 layers of 1 mm Cu wire) was used. The chemical composition of ADI cast iron was Fe-3.7C-0.9Mn-2.3Si-0.7Ni-0.014S (wt. pct.). ADI cast iron samples were austenitized at 920 °C for 30 min. After austenitizing the samples were cooled in sodium/potassium nitrate salt bath. The experimental conditions are presented in Tab. 1. The maximal cooling time in salt bath was 60 seconds and thereafter the samples were moved into EMS sensor which was placed in a furnace. Temperature in the furnace was controlled by a PID regulator. Temperature of the sample was measured by K-type thermocouples which were embedded with the samples where it was connected to a Nanodac data logger (co. Eurotherm). Inductivity of the samples was measured by Agilent U1731C LCR meter (accuracy 0.2%, 1 kHz frequency) and data were saved on the PC (Fig. 1). Further analyses were developed by an algorithm of synchronizing time from temperature data logger and Agilent LCR meter in SciLab 5.4.1 (Scilab Enterprises 2014).

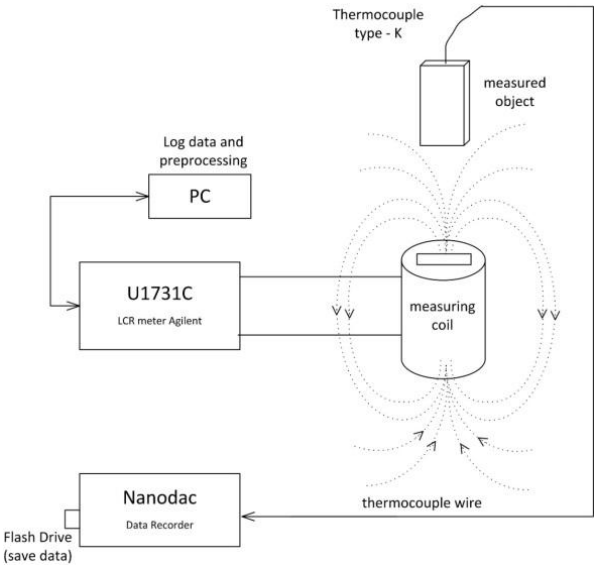


Fig. 1. – Schematic illustration of experimental set-up

Tab. 1. – Measured sample parameters from the experiment

Sample no.	Temperature (°C) PID/sample	Austempered time (min.)	Secondary cooling in	Inductance of the sample at temperature before austenizing (ΔH)
1	200	240	Air	415
2	250			417
3	300			420
4	350			425
5	400			433
6	450			438
7	500			442

Metallographic samples were cut and polished from heat treated samples and etched in a solution obtained by dissolving nitric acid (2 ml) in ethanol (100 ml) Nital. The nital etchant was used for the determination of bainite volume. Austenite phase was determined by using Klemm etchant (2g $K_2S_2O_5$ + 100 ml supersaturated $K_2S_2O_3$ in water) where austenite phase was not attached and bainite or martensite showed dark blue colour. Phase percentage in each sample was measured using SciLab Image and Video Processing toolbox, where threshold was obtained by a binary matrix algorithm. The algorithm was written for a proportion phase's evaluation.

Equation (1) expresses the change of inductance ΔH which is proportional to the change of ferrite volume ΔV_{FB}.

$$\Delta H = \Delta V_{FB} \times F(H)^{-1} \quad (1)$$

Where $F(H)$ is the relationship between inductance (-) and ferrite volume percentage. The study conducted by (YIN ET AL. 2007) shows that $F(H)$ depends on frequency. The relationship between inductance and ferrite can be described by equation (2) which has

been previously determined in published study involving fine ferromagnetic pure iron powder.

$$F(H) = 0.48 \times \frac{H_{act}-H_{ec}}{H_A-H_{ec}} + 0.19 \times \left(\frac{H_{act}-H_{ec}}{H_A-H_{ec}}\right)^2 \; if \; \frac{H_{act}-H_{ec}}{H_A-H_{ec}} \in \langle 0, 0.921)$$

$$F(H) = 5 \times \frac{H_{act}-H_{ec}}{H_A-H_{ec}} - 4 \; if \; \frac{H_{act}-H_{ec}}{H_A-H_{ec}} \in \langle 0.921, 1\rangle \quad (2)$$

Where $H_{act} \in [H_{ec}, H_A]$ is the actual inductance, H_{ec} is the empty coil inductance, H_A is the inductance of the sample at temperature before austenizing.

Two austempering stages have been identified due to different transformation products. The products of the first stage are small bainite ferrite plates with high carbon austenite. High concentration of carbon is produced from the rejection process as the bainitic ferrite grows towards the untransformed high carbon austenite. The second stage of transformation produces the transformation of metastable high carbon austenite to ferrite and ε-carbide, or cementite during long austempering times. The paramagnetic high carbon austenite causes the inductance not to be equal to H_A. This drawback was taken into account in the Avrami equation (3) as the coefficient of $1 - A_{ret}$.

$$V_{FB} = (1 - A_{ret}) \times \left(1 - e^{-k \times t^n}\right) \quad (3)$$

Where A_{ret} is volume of retained austenite, k is coefficient for an initial nucleation time, t is time and n is coefficient for the saturation process of the transformation austenite. The measured data was fitted by Marquard-Lavenberg method, where coefficients k and n were obtained. Value A_{ret} was determined by an optical metallography of the sample which was austempered, and can also be determined by coefficient $1 - A_{ret}$ from Marquard-Lavenberg measured data. Martensite transformation can be detected by electromagnetic sensor as well as martensite temperature.

RESULTS AND DISCUSSION

In Tab. 2 is presented the experimental results. Inductance difference was calculated from the values of empty coil inductance, inductance with heat treated sample at austempered temperature and inductance of the sample at austempered temperature. The results indicate that austempered temperature influenced the maximal value of sample inductance and value of the inductance difference due to untransformed paramagnetic austenite phase. Light optical metallography revealed that samples microstructure contains bainite, martensite and austenite (Fig. 2). Based on theoretical assumption, the value of the inductance difference should be directly proportional to volume of austenite phase in microstructure. The experimental procedure assumed that austempered time (350 min.) should be less than the total transformation time of the austenite phase. The austempered time was set for the first stage where bainitic ferrite and high carbon austenite coexisted at limited time. Cooling of the specimen in air after austempering permitted the transformation of the austenite to martensite. Hence, volume of the austenite phase at austempered temperature before cooling was given as sum of the martensite and austenite phase microstructures.

Tab. 2. – Samples measured parameters

Sample no.	Inductance H_A of the sample at temperature after austenizing (ΔH)	Inductance difference (%)	Volume of austenite (%) in the matrix (Klemm)	Volume of bainite (%) in the matrix (Nital)	Volume of martensite (%) in the matrix – balance
1	392	15	15 ± 2.6	62 ± 4.8	23
2	391	17	16 ± 3	63 ± 4.9	21
3	387	21	15 ± 2.8	59 ± 3.4	26
4	393	20	13 ± 2.9	61 ± 3.6	26
5	385	28	21 ± 3.5	62 ± 4.1	17
6	413	30	25 ± 2.1	57 ± 4.5	18
7	429	7	12 ± 1.8	78 ± 5	10

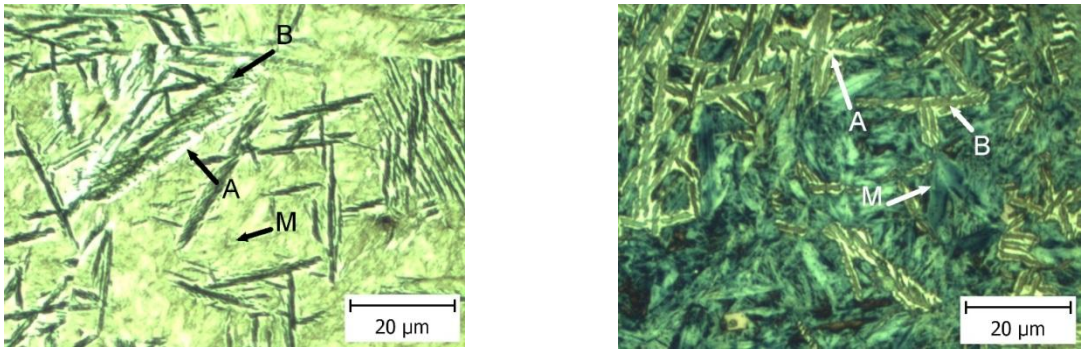
The relationship between inductance difference and volume of the austenite shows that austenite volume is not directly proportional to the inductance difference. This confirms the metallographic method where austenite phase is partially etched by Klemm etchant.

The use of an electromagnetic sensor in isothermal treatment for detection of austenite transformation was monitored by applying Eq. 3. Four samples were respectively austempered at 250°C, 300°C, 350°C and 400°C and samples quenched in water. Austempered

time was equal to $H(\%) = 55 - 60$. At specific temperature of 350°C the same samples were austempered and quenched in water. Austempered time was equal to $H(\%) = 10;\ 25;\ 35$ and 55 . Microstructure of the samples contains bainite, martensite and retained austenite. The relationship between volume of bainite and inductance H (%) is shown in Fig. 3.

Since the evaluation process of microstructure was similar to sample no. 1-7, the threshold errors and standard deviation of mean values of bainite phase was relatively high. The results show that the inductance corresponded with volume of bainite but limit of saturation (62 vol. % of bainitic ferrite) showed high errors. Lower volume of the bainite phase corresponded with inductance with high correlation efficiency (R^2 = 0.97). The correlation between inductance and ferromagnetic ferrite greater than 62 vol. % was not possible due to high deviation by the metallographic method for evaluation of austenite transformation phase products.

Actually volume of bainite phase at austempered temperature was used for determination of time temperature diagram for ADI cast iron. TTT diagram was developed by fitted measured values using Eq. 3. Determination of the martensite temperature was by electromagnetic sensor where the sample was cooled at 200°C in salt bath and further cooled by air in electromagnetic sensor at room temperature. Martensite transformation of the austenite was independent on time but volume of martensite phase was dependent on temperature. The relation dH/dT showed a peak of the actual temperature during the austenite to martensite transformation.



a) b)

Fig. 2. – Microstructure of the ADI cast iron at H (%) = 35; a) etched by Nital, B-bainite, A-austenite, M-martensite b) etched by Klemm

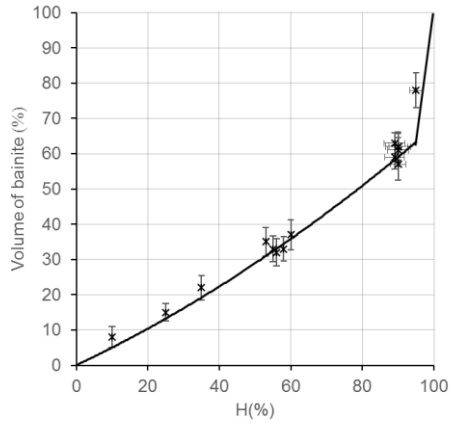


Fig. 3. – Relationship between volume of bainite and inductance (%); line represent Eq. 2

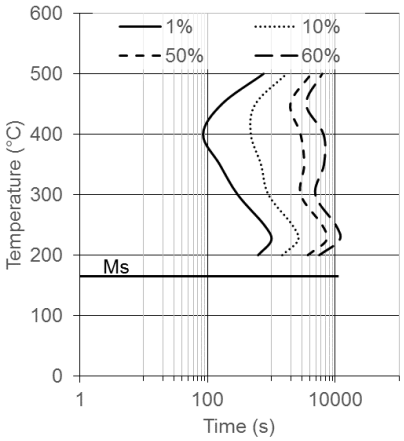


Fig. 4. – TTT diagram developed by results from EMS sensor

CONCLUSIONS

Electromagnetic sensor was suitable for detection of ferromagnetic phase in iron alloys due its sensitivity and good relationship with ferrite phase percentage in austempered cast iron. The correlation efficiency was found to be 97%. Ferrite percentage greater than 62 vol. % decreased the sensitivity of the electromag-

netic method. In addition, electromagnetic sensor detection of the austenite phase transformation at constant temperature could also be used for the description of kinetic austenitic transformation. The device could be used for the investigation of multi-phase iron alloys of different ratio microstructure phases.

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