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Effects of Two Cycle Heat Treatment on the Microstructure and Hardness of Ductile Iron

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ABSTRACT

Austempering is one of the trendiest heat treatment processes to promote the strength and toughness of ductile iron. However, such practice is complex because it involves using aqueous solutions as quenchant (salt bath solution). This study was conducted to analyse the heat treatment of the combination processes of annealing-austenitising and evaluate the correlation between microstructure constituent and hardness of the ductile iron. Ductile iron samples in form of double cylinder was produced by conventional CO_2 sand casting method. The new heat treatment process was started by annealed at 873 K for 1.8 ks before being oil quenched. Subsequently, the samples were austenitised at austenitising temperatures 1123 K, 1173 K and 1223 K for 3.6 ks respectively before being immediately oil quenched to room temperature. A series of microstructure analysis tests, including optical microscopy and X-ray diffraction (XRD) was applied. Vickers microhardness tester was used to measure the hardness for each microstructure constituent. The results showed that ductile iron matrix transforms to martensitic during heat treatment of annealing-austenitising combination processes, which in turn contributes to increasing microhardness of martensite and the bulk hardness of ductile iron

Keywords: Ductile iron, hardness, heat treatment, microhardness

INTRODUCTION

Ductile iron is an engineering material with good mechanical properties due to its microstructural control (Fatahalla, AbuElEzz, & Semeida, 2009; Nabuo et al., 2003). There has been many attempts to quantitatively and qualitatively diversify the microstructure appearance either by adding alloying elements or heat treatment (Susil et al., 2006; Yusuf et

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al., 2010; Salman et al., 2007; Kiani-Rashid & Edmonds, 2008). According to Yusuf et al. (2010), surface and hardness of ductile iron will be improved by combining conventional boronizing and tempering processes. Previous researcher, have added a new sub-cycle called preheat process into conventional austempering cycle (Hsu et al., 2009). Abdullah (2011) also used the similar manner where it was combined with conventional tempering process to improve the mechanical properties of niobium alloyed ductile iron (Abdullah., 2011). Konecná et al. (2013) combined oil quenching and austempering methods in a cycle as new heat treatment practice to heat treat ductile iron (Konecná et al., 2013). Sahin et al. (2010) and Ayman et al., (2009) introduced the new two-steps austempering process where it consists of two stages of austempering process instead of one stage in conventional practice (Sahin et al., 2010; Ayman et al., 2009). The latter is preferred because of the cost and complexity of adding the alloying elements (Chang et al., 2008).

The awareness that heat treatment can improve the physical and mechanical properties of ductile iron this study considers the combination of modified annealing, austenitising processes. It will examine the matrix compositions and transformations in order to evaluate the correlation between microstructure constituent and material hardness on each subsequent cycle in the heat treatment process.

METHOD

Experimental Procedure

The metals used in the present study were made in the laboratory induction furnace with high purity raw materials such as pig iron, carburizer and steel scraps. At 1450°C the molten metal was poured into a 60 kg capacity preheated ladle. The Special alloy recognized as Ferrosilicon Magnesium was used as nucleating agent to nucleate graphite in spherical form. 1.6 wt % of this alloy was placed in treated ladle. The sample was allowed to cool at room temperature before the moulds were broken to get the sample form the double cylinder with dimension of 300 mm long and 25± 2 mm by diameter. The chemical compositions of ductile iron samples were studied with a spectrometer test and results shown in Table 1. Samples were cut into small pieces and initially annealed at 773 K and 873 K in a tubular furnace before being oil quenched. All the samples were continuously austenitised at three different austenitising temperatures which were 1123 K, 1173 K and 1223 K for 3.6 kilo seconds respectively. The samples were then immediately oil quenched to room temperature.

Characterization of microstructure constituents nucleated in the as-cast and heat-treated condition were carried out by optical microscopy. 2% Nital etchant was applied before microstructure was observed and characterized under Olympus BX 41M optical microscope. IMAPs 4.0 edition software was used to capture the presented microstructure. The quantification of the volume of retained FCC austenite, BCC ferrite and BCT martensite were undertaken by X-ray diffraction (XRD) in an Ultima IV diffractometer using Cu-kα radiation in a range

between of 20–120° (Sahin et al., 2010). Vickers Microhardness tester is used in this study to measure hardness of each microstructure constituent. Indentation was employed by applying 25 g load on each microstructure constituent.

Table 1
Chemical composition of ductile iron sample

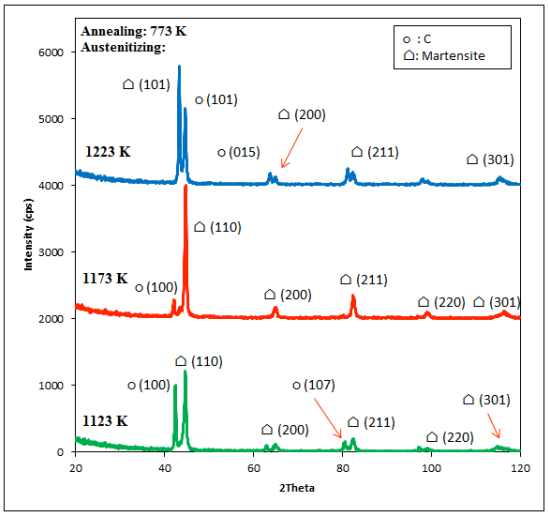
C	Si	Mn	P	S	Cu	Mg	Fe
3.49	2.62	0.55	0.069	0.0074	0.0072	0.014	Balance

RESULTS AND DISCUSSION

XRD

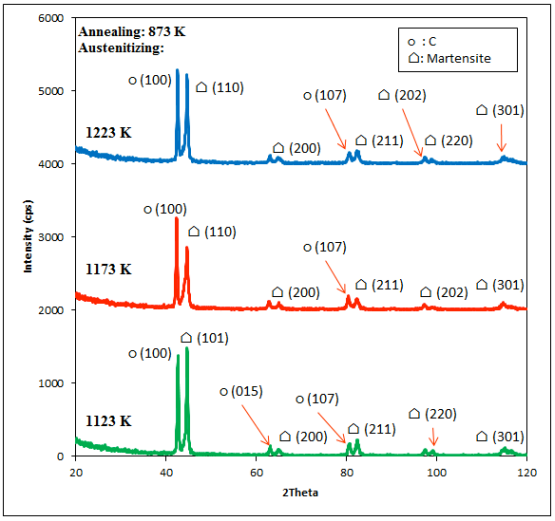
The planes indicated in the XRD pattern of as-austenitised samples annealed at 773 K, as shown in Figure 1. The peaks of martensite are also identified at 2θ angle 44° with the planes (101) and (110) in the XRD patterns of as-austenitised samples annealed at 873 K, as indicated in Figure 2. The presence of both planes also suggests that BCT martensite is the main constituent for the as-austenitised samples annealed at 773 K and 873 K. Annealing process does not influence the phase in the as-austenitised samples even the samples were annealed to the elevated temperature which below than critical eutectoid temperature. Despite annealing process was initially changed in quantitative the phase constituents such as pearlite and ferrite in the first stage of heat treatment, but it shows no effect yet when the sample was further austenitised to critical eutectoid temperature. Aside of the martensite, a graphite peak is likewise presented in the planes (100) and (101) between 2θ angles of 42° and 44°, in all cluster of samples which was austenitised at different temperatures. Graphite peaks are present in the XRD patterns for all samples because carbon is one of the main alloying elements in producing ductile iron (Chang et al., 2008).

Based on the XRD patterns shown in all clusters of as-austenitised sample, martensite transforms at austenitising temperature of 1123 K (Chang et al., 2008). Austenite generally nucleates when the temperature rises beyond the critical eutectoid temperature, so too were samples austenitised at 1173 K and 1223 K (Sahin et al., 2010). Rao and Putatunda (2003) stated that volume fraction and carbon content of austenite increases when austenitising temperature rises beyond the critical eutectoid temperature (Rao & Putatunda, 2003) as carbon atoms trapped in the crystal structure when austenite are diffused during heating.



Graphite, C – (ICDD: 261079); Martensite, α'-Fe – (ICDD: 441289)

Figure 1. XRD pattern of as-austenitised ductile iron (annealed: 773 K) at different austenitising temperatures



Graphite, C – (ICDD: 261079); Martensite, α'-Fe – (ICDD: 441289)

Figure 2. XRD pattern of as-austenitised ductile iron (annealed: 873 K) at different austenitising temperatures

Microstructure

The annealing process did not influence the appearance of as-austenitised microstructure as shown in Figure 3 and Figure 4 where the samples were initially annealed at 773 K and 873 K respectively before austenitised at 1123 K, 1173 K and 1223 K. Figure 3(a) and Figure 4(a) depict the microstructures of as-annealed ductile iron at temperatures of 773 K and 873 K respectively. The microstructures of as-annealed sample consist of typical microstructure constituents which contains graphite nodules, the sea of ferrite (white region) and the island of pearlite (dark region). The microstructures are almost similar qualitatively with as-cast microstructure where graphite nodules are surrounded by ferrite and pearlite.

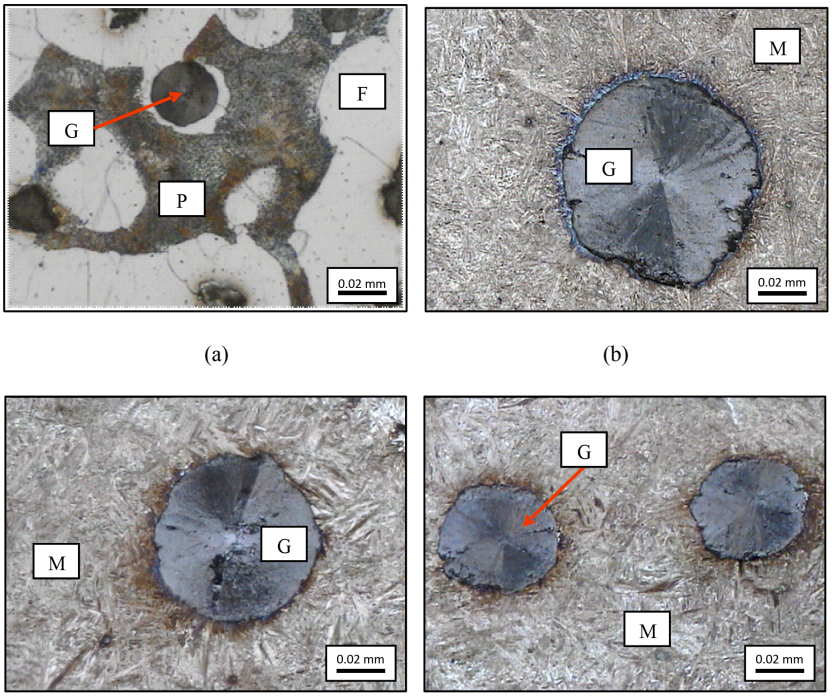


Figure 3. Microstructure of As-Austenitised Ductile Iron (Annealed: 773 K) at Different Temperatures: (a) As-Annealed; (b) 1123 K; (c) 1173 K; and (d) 1223 K. Note: G is Graphite; F is Ferrite; P is Pearlite; M is Martensite

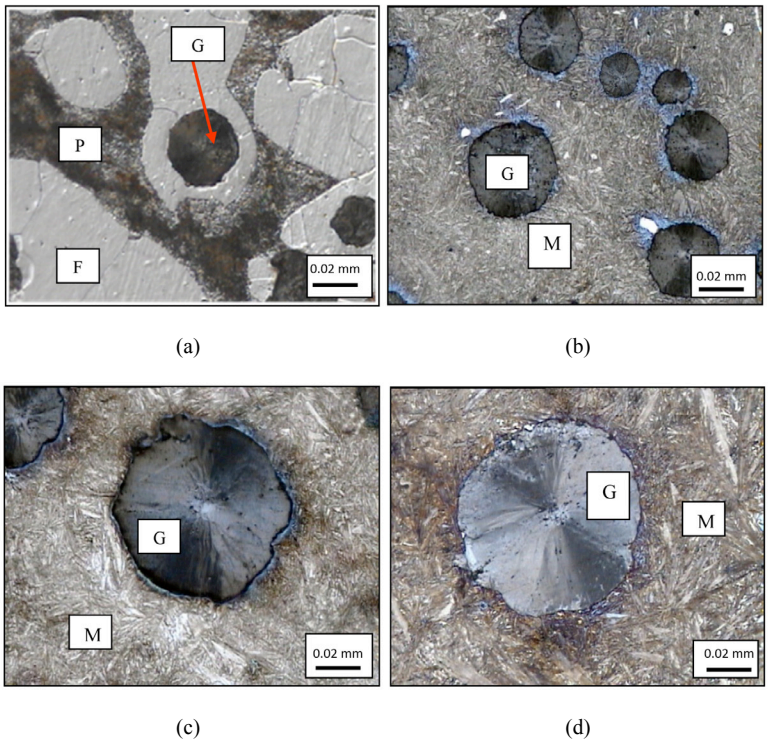


Figure 4. Microstructure of As-Austenitised Ductile Iron (Annealed: 873 K) at Different Temperatures: (a) As-Annealed; (b) 1123 K; (c) 1173 K; and (d) 1223 K. Note: G is Graphite; F is Ferrite; P is Pearlite; M is Martensite

Martensite is remarkably transformed because austenite nucleated during the austenitising process was rapidly cooled to room temperature via oil quenching. Austenite with low residual carbon content trapped in the crystal structure did not have time to stabilize and diffuse out of the crystal structure (Kiani-Rashid & Edmonds, 2009), leading to its transformation into martensite at room temperature. The lath-shaped crystal grains of martensite became coarser when austenitising temperature increased till 1223 K (Rao & Putatunda, 2003) resulting from the volume fraction and carbon content of austenite which increased when austenitising temperature rises beyond the critical eutectoid temperature (Rao & Putatunda, 2003).

Hardness

Figure 5 shows the average values of Vickers microhardness of martensite and graphite structures of as-austenitised ductile iron annealed at 773 K and 873 K. Hardness of martensite increased gradually when austenitising temperature increased till 1223 K. on the other hand, the average graphite hardness values remained constant at range of 38 HV to 39 HV. The microhardness trends of martensite and graphite show no changes though annealing

temperature increases up to 873 K. Nevertheless, the average martensite hardness values of the samples slightly increased as a result of greater carbon diffusion in the matrix when annealing temperature is raised.

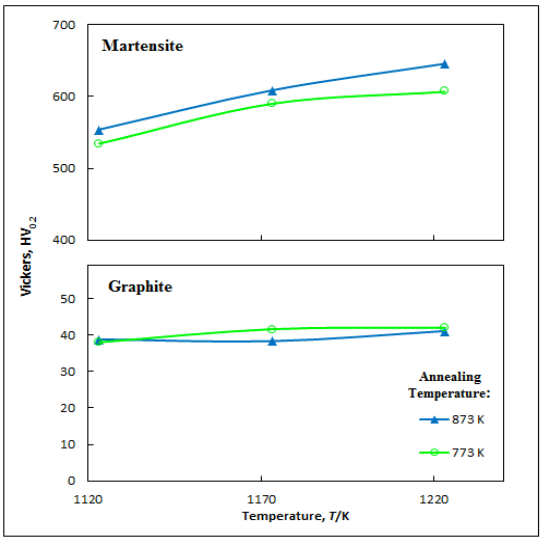


Figure 5. Vickers Microhardness (Load: 0.2 N) of Martensite and Graphite at different austenitising temperatures (Annealed: 773 K and 873K)

Graphite is less hard compared to Martensite. Graphite nodules presented in the matrix do not contribute to the precipitation hardening though all clusters of sample have been austenitised beyond critical eutectoid temperature. Furthermore, the increase in Martensite hardness as function of austenitising temperature is caused by carbon precipitation and enrichment of the matrix constituents (Kiani-Rashid & Edmonds, 2009; Rao & Putatunda, 2003).

CONCLUSION

The new heat treatment process, combination of annealing and austenitising process, found that ductile iron matrix transforms to martensitic instead of ferritic-pearlitic matrix shown. Austenitising temperature does slightly influence the martensite morphology, and increasing it results in the increasing of microhardness of martensite and the bulk hardness of ductile iron.

In conclusion, microstructure constituents have good correlation with hardness of ductile iron. The type of microstructure and hardness of each constituent require consideration to effectively improve the hardness of ductile iron. Proven that, this hardness could be estimated using mixture law of hardness for each microstructure constituent.

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