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EFFECT OF DOUBLE-QUENCHING HEAT TREATMENT PROCESS ON MICROCONSTITUENT AND XRD OF DUCTILE IRON

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ABSTRACT

Heat treatment is one of the preferred methods in altering material characteristic. In this present study, the effect of austenitizing temperature in double-quenching heat treatment process was studied on microstructure as well as exploring the phase constituents of ductile iron through x-ray diffraction(XRD)analysis. Ductile iron produced through conventional CO_2 sand casting method. Ductile iron was heat treated by using the double-quenching method. The sample was annealed at 673K for 1.8 ks and subsequently oil quenched before austenitized at 3 different temperatures which those are 1123K, 1173K and 1223K for 3.6 ks respectively. Standard metallographic observation nodule and XRD analysis were done to characterize the microstructure and the constituents respectively.It is found that austenitizing process transforms the microstructure from pearlitic-ferritic matrix structure, which presented in annealing condition into martensitic. Martensite morphology becomes coarser and the nodularity of graphite nodule decreases as austenitizing temperature increases. But, austenitizing process seemingly gives no significant effect to the graphite nodule size. The presence of BCT martensite is validated by the presence of high-intensity (110) and (101) planes verified at 43° to 44° 2θ angles. Austenitizing sub-process of double-quenching heat treatment process does influence the martensite and graphite nodule morphologies. It transforms ferritic-pearlitic structure in as annealed ductile iron to martensitic matrix structure. The presence of Martensite and austenite can be validated by using XRD method with specific plane.

Keywords: new heat treatment, martensite, austenitizing, nodule analysis.

INTRODUCTION

Ductile iron which is one of the most preferred materials in engineering applications as well as manufacturing industries nowadays is a part of the cast iron family. Ductile iron is also known as nodular or spheroidal graphite cast iron because the physical appearance of graphite embedded in the microstructure of ductile iron reveals in shape of uniform tiny spheres [1-4].

Generally, most of engineering properties of a material are closely related with the matrix structure[5].Hence, heat treatment process has nowadays become more preferable and essential tools in metal fabrication like cast iron and steels[6,7]. Heat treatment conditions like holding temperature and period, cooling medium and the atmospheric environment essentially influence the microstructure nucleation and also mechanical properties of ductile iron [8-12].

Past researchers found that the carbon content in the matrix of austempered ductile iron (ADI) increased but decreased that of free ferrite when austenitizing temperature increased [13-15]. Furthermore, in [16] developed a new heat treatment process to produce a dual matrix structure of ductile iron. They found that the matrix structure does influence the abrasive wear behavior of ductile iron. In [17] proved that the formation of free-carbide bainite structure can be controlled by the addition of alloying element, aided by the heat treatment process. In addition, in [18] reported that the phase amount of partially-austempered ductile iron is affected by the austenitizing process.

Realizing about the great effect provided by heat treatment process on the microstructure and mechanical properties of ductile iron, a new heat treatment process being introduced in this study where it comprises a combination of modified annealing and austenitizing processes. By combining these heat treatment processes in a single cycle, the advantages in each heat treatment process are expected could be combined [19]. As the author concerns, lack of published research discussed comprehensively about the effects of this heat treatment combination on microstructure constituents and mechanical properties of ductile iron.

METHODOLOGY

Preparation of sample

The samples used for this study were prepared in the form of double cylinder with dimension of 300mm long and 25±2mm diameter both using the CO_2 sand casting method. Ferrosilicon magnesium has been used as nucleating agents. The chemical compositions of the samples obtained through spectrometer test are shown in Table-1.

Table-1. Chemical composition of ductile iron sample.

Element	DI
C	3.49
Si	2.62
Mn	0.55
P	0.069
S	0.0074
Fe	Balance

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Heat treatment

Double-quenching heat treatment method was carried out to the samples. Samples were initially annealed at 673 K for 1.8 kilo seconds before being oil quenched. Then, all the samples were continuously austenitized at 3 different austenitizing 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.

Microstructure and XRD analysis

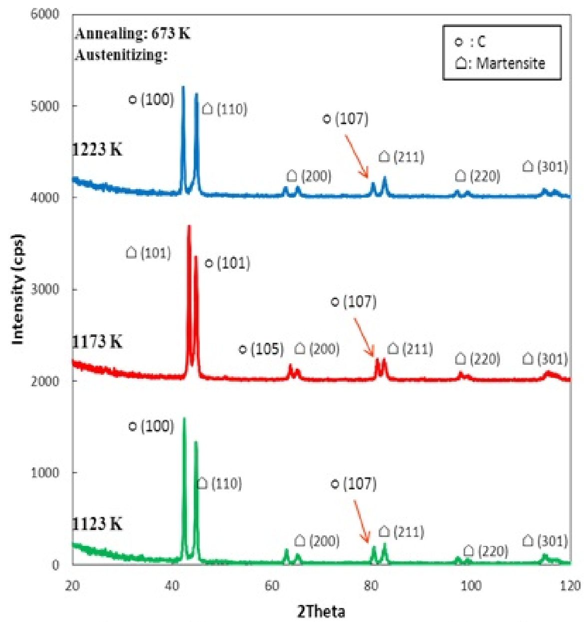
Identification of the structure presented in the samples was determined by a series of standard microstructure observation method after etched with 2% nital. The graphite nodules were analyzed by applying Leica Image Analyzer and Software. The average nodule size of the sample is approximately 24 µm. X-ray diffraction (XRD) analysis was performed at 40 KV and 40mA using copper Kα radiation to analyze the phases presented. The Rigaku rotating anode head diffractometer has been used to scan on an angular range between 20˚ to 120˚ at scanning speed of 1˚/minute. The data were analyzed using integrated powder difraction (PDXL) software to interpret the peaks such as face centered cubic (FCC)austenite, body centre cubic (BCC)ferrite and body center tetragonal (BCT) martensite.

RESULTS AND DISCUSSION

XRD analysis

Error! Reference source not found. shows the XRD patterns of as-austenitized sample at different temperatures, which were firstly annealed at 673 K respectively. The presence of high-intensity (110) and (101) planes verified at 43° to 44° 2θ angles suggests that the BCT martensite is the main constituent. Annealing process does not influence the phase in the as-austenitized samples, even the samples were annealed to the elevated temperature which below than critical eutectoid temperature. However, it might affect the carbon precipitation in the phase and grain refinement. Aside of the martensite, a graphite peak is likewise presented in the planes (100) and (101) between 2θ angles of 42° and 44°. Graphite peaks are present in the XRD patterns for all samples because carbon is one of the main alloying elements in producing ductile iron.

Martensite transforms at the austenitizing temperature of 1123 K because the temperature applied is higher than the critical eutectoid temperature. Austenite generally nucleates at the temperature rises beyond the critical eutectoid temperature. The higher the austenitizing temperature, the more carbon diffuses which it affects the microconstituent characteristic. Then, the martensite would be transformed when austenite rapidly cools to room temperature via oil quenching. It similarly occurred to those samples austenitized at 1173 K and 1223 K because the temperatures applied are higher than the critical eutectoid temperature. In [14] stated that volume fraction and carbon content of austenite increase when austenitizing temperature increases rises beyond the critical eutectoid temperature. Carbon atoms diffused during heating were trapped in the crystal structure when austenite rapidly cools to room temperature, thus producing martensite crystal structure.



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

Figure-1. XRD pattern of as-austenitized ductile iron at different austenitizing temperatures.

Microstructure

Error! Reference source not found.(a) depicts the microstructure of the as-annealed ductile iron at annealing temperature of 673 K. While **Error! Reference source not found.**(b-d) show the microstructure of as-austenitized ductile iron at different austenitizing temperatures which were at 1123 K, 1173 K and 1223 K. It demonstrates that austenitizing process in this double-quenching process changed the microstructure from pearlitic-ferritic matrix structure into martensitic, as verified in XRD patterns. Annealing process did not influence the appearance of as-austenitized microstructure because the similar structures are also observed when ductile iron initially annealed at different temperatures. The microstructures of as-annealed sample consist of typical microstructure constituents where includes of 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.

Austenite with low residual carbon content trapped in the crystal structure has no sufficient time to stabilize and diffuse out of the crystal structure. Therefore, the austenite transformed to martensite at room temperature. The lath-shaped crystal grains of martensite

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become coarser when the austenitizing temperature increased till 1223 K. in [14] also acquired that the microstructure of ADI became coarser when the austenitizing temperature increased. The change of microstructure morphology is owing to the volume fraction and the carbon content of austenite, which increased when the austenitizing temperature rises beyond the critical eutectoid temperature.

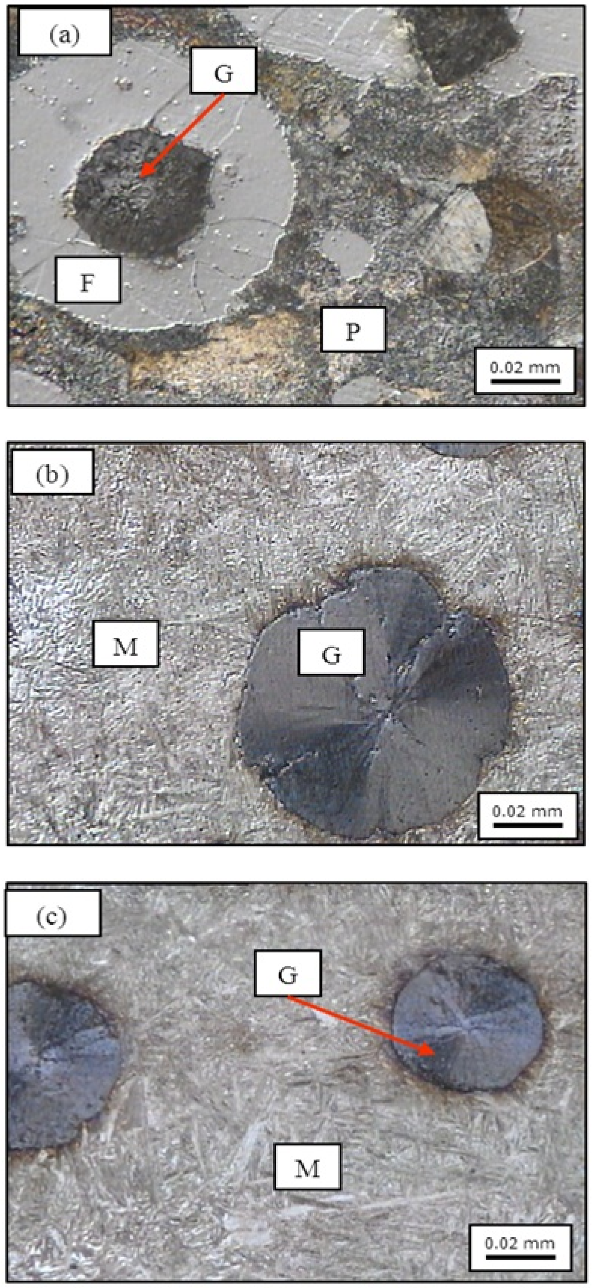
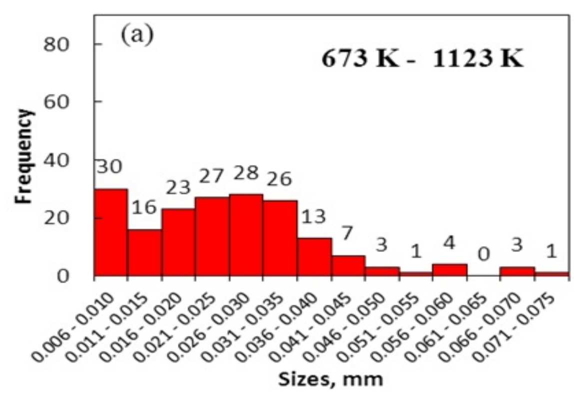


Figure-2. Microstructure of as-austenitized ductile iron at different temperatures: (a) As-annealed; (b) 1123 K; (c) 1173 K; (d) 1223 K. G is graphite; F is ferrite; P is pearlite; M is martensite.

Nodule analysis

Figure-3 indicates the distributions of nodule with the function of size for the samples austenitized at 1123 K, 1173 K and 1223 K. Nodule distributions showed by the samples are different with each other, especially the number of nodules counted with the function of size. Nodule distribution of as-austenitized samples at 1123 K and 1223 K is more uniform rather than sample austenitized at 1173 K. While, normal nodule distribution (bell-shaped) is shown by the sample austenitized at 1173 K. In this sample, the nodules with sizes between 0.021 mm to 0.025 mm have the highest number of nodules embedded in the matrix.

Figure-4 indicates the effect of austenitizing to the size and nodularity of the nodular graphite, as the samples were initially annealed at 673 K. Austenitizing seemingly gives no significant effect on the average nodule graphite size. The average nodule graphite size of this cluster of samples is approximately 24 µm. Austenitizing process seemingly influences the nodularity of samples. The nodularity is slightly decreased as austenitizing process applied and the temperature increased up to 1223 K. Nodularity of nodular graphite changed because of matrix intrusion during the heat treatment process. in [11] reported that graphite was precipitated on the graphite nodule during the heat treatment process because of carbon migration from the matrix. Therefore, there are changes in nodularity.



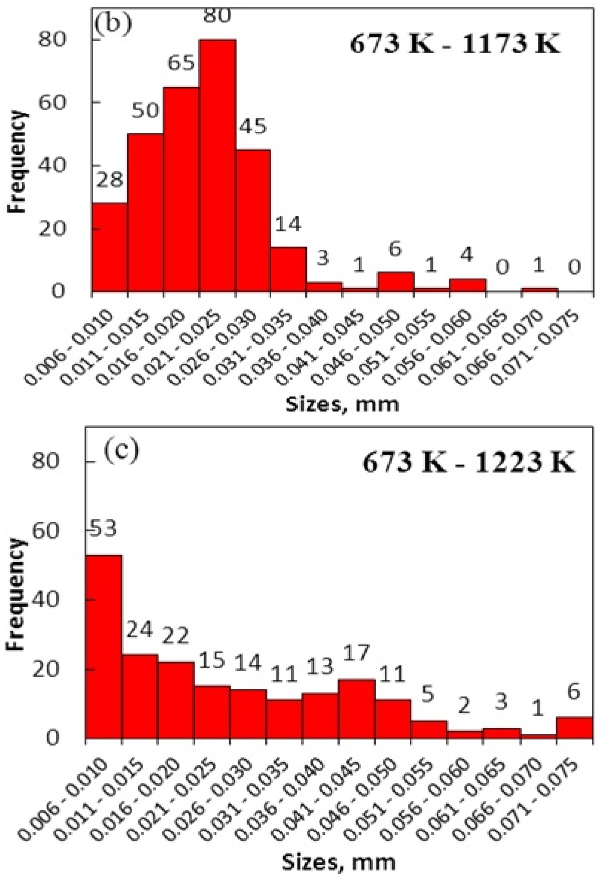
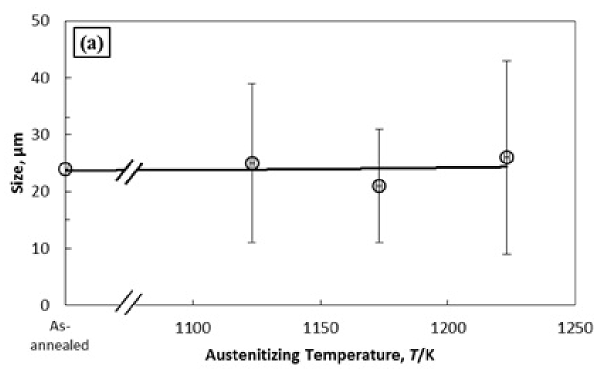


Figure-3. Nodule distribution of as-austenitized ductile iron at different temperatures: (a) 1123 K; (b) 1173 K; (c) 1223 K.



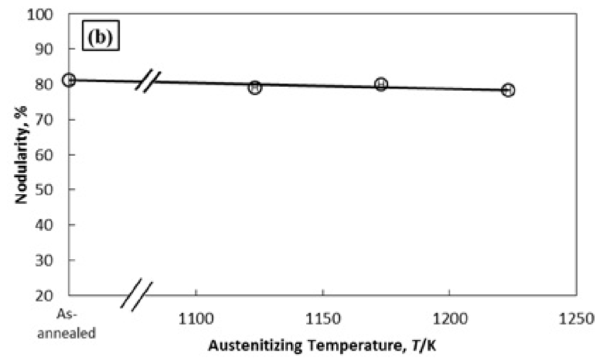


Figure-4. Relationship between austenitizing temperature and: (a) average nodule size; (b) nodularity.

CONCLUSIONS

In the double-quenching heat treatment process, ductile iron matrix transforms to martensitic instead of ferritic-pearlitic matrix shown in the as-annealed sample. Austenitizing temperature does influence the martensite morphology. The presence of BCT martensite is validated by the presence of high-intensity (110) and (101) planes verified at 43° to 44° 2θ angles. Austenitizing process also does influence the nodularity of graphite nodule.

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