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Effect of Heat Treatment (Ferritizing) on Chemical Composition, Microstructure, Physical Properties and Corrosion Behaviour of Spheroidal Ductile Cast Iron

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Two steps ferritizing technique was applied on ductile cast iron samples by austenitizing at 900ºC, air cooling to produce pearlite, ferritizing by reheating samples for different times at 700ºC and air cooling to room temperature. Chemical analysis and microstructure showed that as ferritizing time increased, an increase of percentage of ferrite, decrease of pearlite, with corresponding decrease in cementite and increase of free carbon in the form of spheroidal graphite. These changes explain the changes of physical (mechanical) properties represented in the increase of percentage elongation, decrease of tensile strength and decrease in brinle hardness. Weight loss corrosion test technique was followed for investigation of corrosion rate of heat treated samples in 0.1 N H_2SO_4 solution, which show decrease in corrosion rate with increased ferritizing time. This was explained due to decrease of cathodic sites represented in cementite forming pearlitic lamella. The exception was in the early step of ferritizing, where the corrosion rate increased due to formation of secondary graphite acting as effective cathodic sites.

Key Words: Ferritizing, Pearlite, Austenitizing, Microstructure, Cementite, Spheroidal graphite, Cathodic, Corrosion, Secondary graphite.

INTRODUCTION

Ductile (nodular or spherulitic graphite) cast iron in which a part or all of the carbon is present in the form of a tiny spherical balls, of average 33 to 37 μm[1-4]. This ductile cast iron shows high strength and appreciable tensile ductility. It is used in many branch of industries, *e.g.*, in automobile and diesel engine production, it is used for mill rolles. In chemical and oil industries, it is used for pumps and globe valves operating with corrosive media. It is also used in manufacturing ductile water pipes and wastewater

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pipes and in agricultural, mining, military and railroad components which traditionally produced by expensive forging process involving high grade alloy steel[1-5].

Ductile cast iron is obtained by making small ladle addition of certain alkali or alkaline-earth metals to the liquid iron. In the majority of cases, the addition consists of 0.03-0.07 % Mg. The presence, of magnesium changes the nucleation and growth of graphite during solidification of the Fe-C eutectic from the formation of flakes to the formation of nearly perfect spherules[5-7].

The following phases of Fe-C alloys are generally designated in microstructure of ductile casting.

Graphite = free carbon in spheroidal form represent most of carbon contents.

Ferrite = solid solution of carbon and other elements in, bcc iron, which can dissolve up to 0.04 % c. Microscopically, ferrite appears as homogeneous grains[8].

Austenite = solid solution of carbon and other element in, FCC iron. This structure can dissolve up to 2.0 % C.

Cementite = iron carbide Fe_3C. It is a chemical compound of iron and carbon which contains 6.67 % C. The crystal structure of cementite is very complicated.

Pearlite = two-phases lamellar structure of alternative ferrite and cementite. Pearlite has higher hardness and lower plasticity than ferrite[5,9,10]. It is formed on decay of austenite[1-3] and is stable below 723ºC. The microstructure of pearlite can be examined by polishing followed by etching with suitable etchant such as nital (3-5 % nitric acid in ethanol). The cementite lamella are cathodic to ferrite lamella with a substantial potential difference, so that smooth anodic dissolution of ferrite lamella with the cathodic cementite lamellas. The cathodic lamella appear bright while the anodic ferrite lamellae is recessed below the original plane of polish and can appear to have a rough surface which will make it appears dark under bright field of illumination[8].

Heat treatment of ductile cast iron

Heat treatment of ductile cast iron can give a wide range of mechanical properties, which are suitable for many applications. When maximum ductility and impact strength along with best machinability are required, ferritizing annealing becomes a necessity. The purpose of annealing is to achieve a structure consisting of graphite nodules in a fully ferritic matrix. In addition, ferritizing annealing assures uniformity of the mechanical properties and the absence of carbides and chill, Ferritizing can be done either on the melt in case of producing (as-cast) ferritic (thin-section) iron castings or on the solid castings[1,11,12] for (thick-section) parts, where an example annealing cycle for ferritizing is two steps annealing consists of:

Austenitizing at temperature (900-940ºC) for times ranging from 1 to 3 h which was a sufficient period to ensure that the matrix is fully transformed to austenite.

Air cooling of austenite where samples transformed to pearlite formation of (ferrite-cementite) lamella.

Reheating to 700ºC for suitable times for transform to ferrite and secondary graphite as

$$Fe_3C \rightarrow 3Fe + C \quad (1)$$

Corrosion of spheroidal graphite cast iron

Gehelehbashi and Davami[13] studied the effect of graphite shape on corrosion which showed that spherodizing reduces the corrosion rate due to reduction of the galvanic potential between graphite and the matrix, which results in production of minimum surface area interface of spheroidal graphite with the matrix[11,13].

The effects of pearlitic structure on corrosion rate were explained[8] as pearlite structure is formed of alternative ferrite and cementite lamella in which cementite acts as cathode to ferrite with substantite potential difference leading to anodic dissolution of ferrite constituent.

EXPERIMENTAL

Specimens material were made of high strength ductile cast iron machined in the form of discs (50 mm diameter and 5 mm thickness) at low machining rate operated in a stream of cooled kerosene to avoid elevation of temperature[6,14]. All chemicals used were of analytical grade. Distilled water used for preparing solutions and samples washing was of conductivity (4×10^{-6} ohm^{-1} cm^{-1}).

Chemical composition of specimens was determined using emission spectroscopic technique with aid of ARL quantmeter as (%) 3.50 C, 0.32 Mn, 0.018 S, 0.042 P, 2.13 Si, 0.082 Mg and 93.88 Fe.

The percentage of graphite carbon was determined by dissolving a weight of the sample in (1:3) nitric acid which dissolved the carbide and left the graphite which separated by filteration, washed, dried and weighed. The combined carbon was determined as the difference between total carbon and the graphite carbon. The cementite F_3C was calculated as equal to = combined carbon × 14.948 (Fe_3C/C).

Heat treatments was carried out in an electric muffle furnace. Charcoal was placed around the sample to avoid any oxidation, two stage annealing cycle was used, is shown schematically in Fig. 1. The austenitizing annealing for 2 h at 900ºC was followed by air cooling to room temperature and immediate reheating (ferritizing annealing) at 700ºC. For different holding times before slow cooling again to room temperature[11,12].

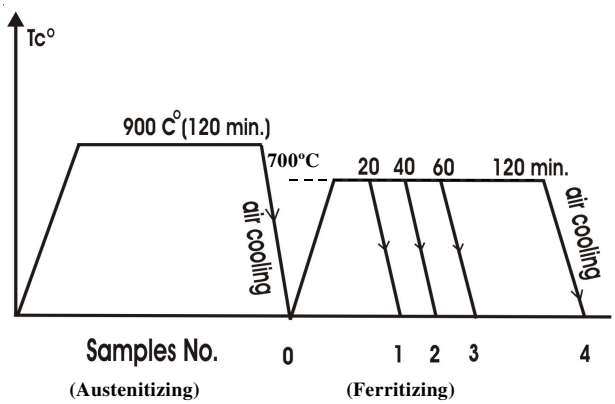


Fig. 1. Scheme of heat treatment of Ductil iron samples

The sample No. 0 without ferritizing and sample No. 1, 2, 3 and 4 were obtained after holding time at 700ºC for 20, 40, 60 and 120 min, respectively. Samples were stress released by reheating to 260º C for 2 h and then left to cool. Microstructure changes in the heat treated samples were characterized by using optical microscope technique[8], after mechanically polished and chemically etched using klemm I etchant for colour etching (product of struers, Copenhagen, Denmark).

Physical properties tests for samples before and after ferritizing were carried out according to ASTM standard[14] using Shimadzu machine (Model UMH-20 Kyoto, Japan) for determining Tensile stress, Elongation percentage and Brinel hardness tests using (Shimadzu-Hardness Machine).

Corrosion measurements

Corrosion behaviour of the heat treated samples (0-4) was studied in aqueous solutions of 0.1 N sulphuric acid at 20, 30 and 35ºC by weight loss technique[15-17]. Specimen (0-4) were polished mechanically using metallurgical emery papers (120, 400, 600 and 800), then rinsed with distilled water, dried and coated with araldite except one circular cross section (to expose to the test solutions), leave for araldite solidification. Samples exposure surface was repolished, rinsed with distilled water, dried and weighed, before and after exposure to test solution for 2 h with mechanical stiring. The specimen was washed with hot distilled water during brushing with hard plastic brush to remove any corrosion products on the surface. The washing water was collected and added to the test solution. Weight loss due to corrosion were determined either directly[15] or by titration[16,18] of dissolved iron using $K_2Cr_2O_7$ (0.01 N) solution, where 1 mL 0.01 N $K_2Cr_2O_7$ = 0.56 mg Fe where true weight loss =

$$\text{weight of Fe} \times \frac{100}{\%\text{ of Fe in the sample}}$$

RESULTS AND DISCUSSION

Heat treatments on the percentage of free carbon (graphite) in samples (0-4) is given in Fig. 2 represent the effect of ferritizing time (minute) on the composition of specimens. Fig. 2 shows the increase of graphite from (2.67 to 3.25 %) with increase of ferritizing time while percentage of cementite (Fe_3C) was decreased from 0.83 % at time 0 to 0.23 % at time 120 min. The increase of spheroidal graphite as ferritizating time increase from 0 to 120 min was explained in the light of the fact that in ferritizing process the cementite Fe_3C was converted to Fe and small spheares of graphite. This resulted graphite was called secondary graphite which diffused and accumulated to the big spheroidal graphite. The micrograph of etched samples (0-4) are given in Fig. 3.

(1) Increase of ferrite phase (white closured) percentage from sample no. 0 to sample 4, respectively the values were indicated in Fig. 2. (2) Corresponding decrease in both pearlitic (red coloured) phase and the % of perllitic phase was given in Fig 2. (3) Graphite is present as balls or spheres dispersed completely in ferritic matrix and remainder pearlite (Fig. 3).

Mechanical (physical) properties

As ferritizing developed by time 0, 20, 40, 60, 120 min for samples No. 0, 1, 2, 3 and 4:

(1) Tensile strength and hardness decreased (Fig. 2). These were explained due to increasing ferrite phase which has moderately good plasticity and strength while the carbide (cementite Fe_3C) which is very hard and brittle decreased.

(2) The elongation which is a measure for ductility increase in the order: sample (0-4) as ferritizing process was developed, the ductility increased due to elimination of secondary graphite and formation of spherulite graphite having a low surface-to-volume ratio.

Effect of ferritizing on corrosion behaviours

The corrosion rate was determined for samples (0-4) using weight loss method[15-18].

Where the corrosion rate (mpy) mils per year = $(K \times W)/(A \times T \times D)$ where K = constant (3.45×106), T is time of exposure in hours A is area in cm^2, W is mass loss in gram and D is the density[16] g/cm^3.

The relation between corrosion rate and solution temperature for samples 0, 1, 2, 3 and 4 are given in Fig. 4.

(1) Increase of corrosion rate with temperature for all samples. (2) At 30 and 35º C a decreasing of corrosion rate as ferritizing was developed from sample 0, 2, 3 and 4 an exception for sample no. 1 which have the highest corrosion rate than full pearlite sample no. 0. This was explained in

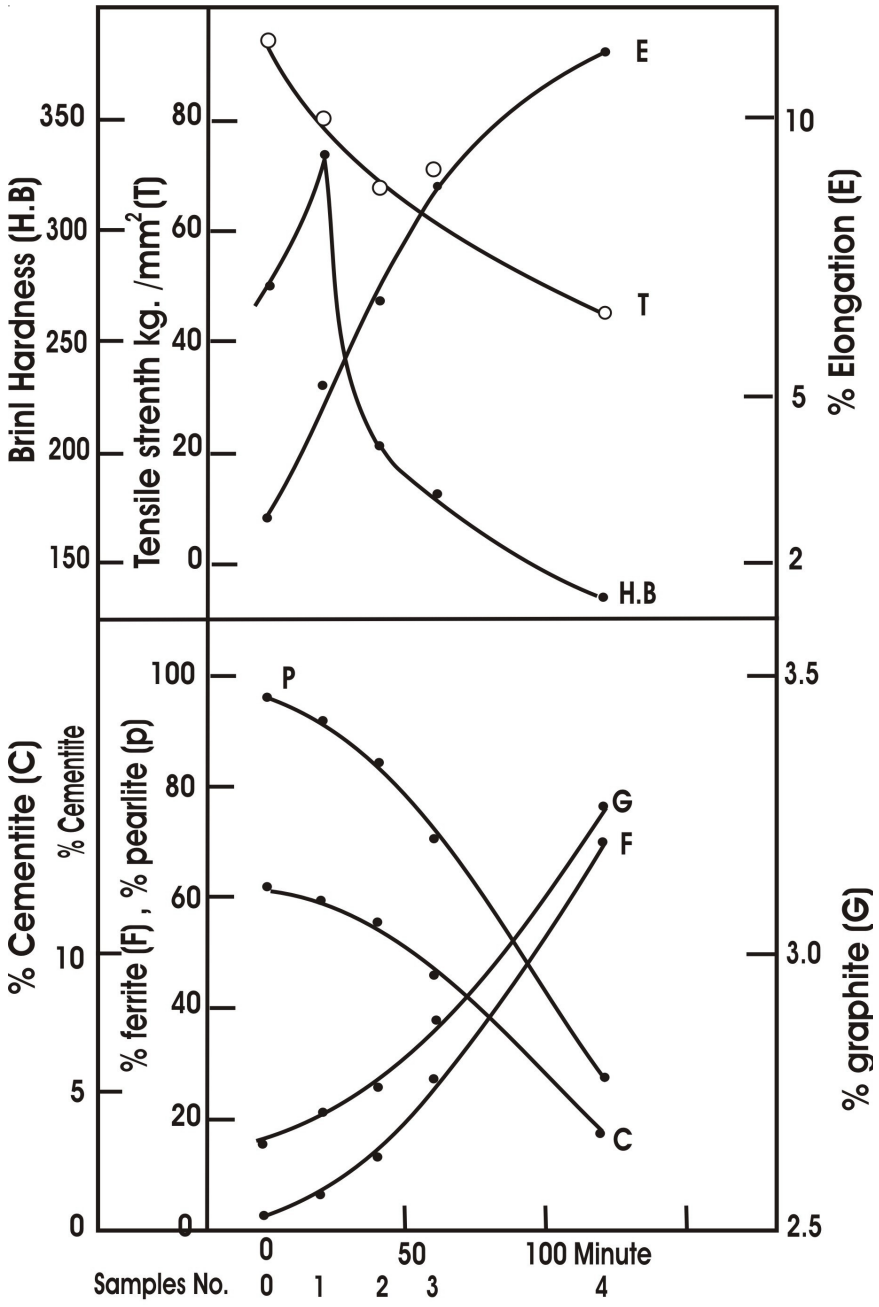
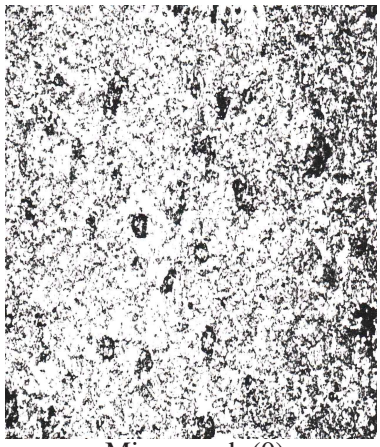
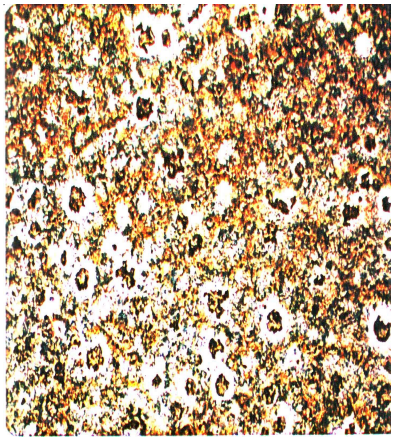


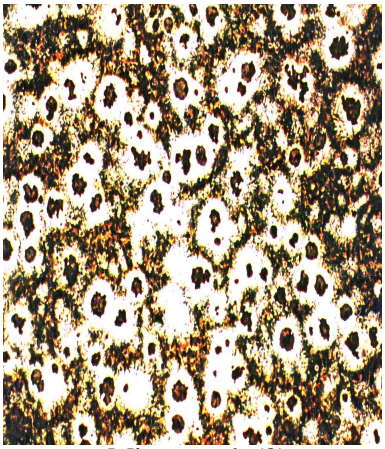
Fig. 2. Values of chemical and structure composition of ductile. Cast iron at different ferritizing times and corresponding physical (mechanical) properties



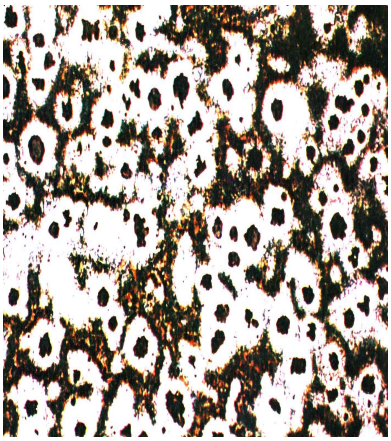
Micrograph (0)



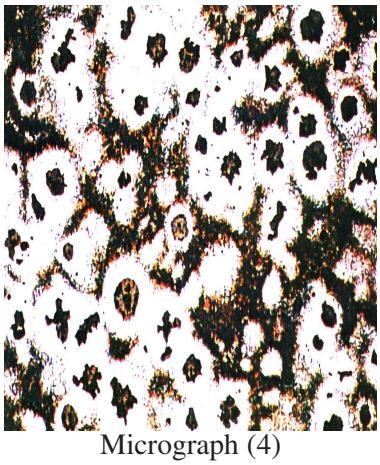
Micrograph (1)



Micrograph (2)



Micrograph (3)



Micrograph (4)

Fig. 3. The micrograph of samples (0-4) etched by klemm I for colour etching (product of struers, Copenhagen, Denmark) which are given in micrograph number 0, 1, 2, 3 and 4, respectively (X300)

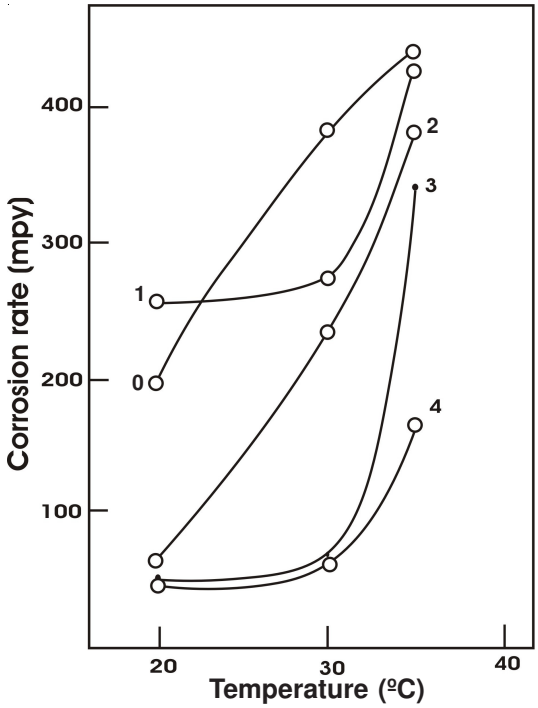


Fig. 4. Values of corrosion rate at different temperatures 20, 30, 35ºC for samples no. 0, 1, 2, 3 and 4, respectively with increasing of ferritizing time

the light of the fact that in the first steps of ferritizing represented in sample 1 in which secondary graphite was formed and spereaded in ferrite, secondary graphite potential is more cathodic than cathodic cementite forming pearlite, leading to stimulate corrosion more than that of pearlite.

As ferritizing process developed in sample no. 2, 3, 4, this secondary graphite diffused and coagulated to larger spheroidal graphite. This graphite addition has very small effect on area to volume ratio of spheroidal graphite and had no significant effect on final corrosion rate.

At 20ºC the secondary graphite cathodic sites was polarized by hydrogen adsorption which stoping its action as cathod so corrosion rate did not increased as at 30, 35º C where this sites were depolarized by temperature and acts as effective cathods.

Conclusion

We can use heat treatment (ferritizing) process of ductile cast iron to get good machinability, increase of percentage elongation, increase of ductility[4] and high corrosion resitance. Specialy in case of drinking water ductile cast iron pipes where it is impossible to use corrosion inhibitors for internal protection of the pipes, at the same time corrosion of the external pipe surface decreases *i.e.*, external protection cost decreases.

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