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The Influence of Austenitizing Temperature and Austempering Time on the Microstructure and Mechanical Properties of Alloyed Austempered Ductile Cast Iron.

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ABSTRACT :*The effect of austenitising temperature and austempering time on the microstructure and mechanical properties of alloyed Ni-Mo-Cu ductile iron was investigated. The specimens were austenitised at 850, 875, 900 and 930^{0}C for 2 hrs, then austempered for 10, 30, 60, 120, 240 and 360 min at 350^{0}C, and examined by optical microscopy. The volume fraction of retained austenite, bainite, martensite and austenite-carbon-content results were then correlated with microstructural changes, the kinetics of transformation and impact toughness. Following short periods of austempering time, the carbon content of the retained austenite is low and on subsequent cooling to room temperature it transforms to martensite, resulting in low impact values. The carbon content of the austenite was evaluated by measuring the lattice parameters using X-ray diffraction. Optimum properties were obtained at 900^{0}C for intermediate austempering periods (120-240 min).*

KEYWORDS: *Austempered Ductile Iron, retained Austenite, Austempering, Austenitising*

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I. INTRODUCTION

Manufacturers are always researches for new alloys which can provide them better advantage at low cost and light weight. To do so, we need to realize the potential of new alloys and relative comparison of their properties. Austempered ductile cast iron (ADI) is relatively a new alloy which is obtained by austempering of ductile cast iron.

Heat treatment of ductile cast iron is carried out in solid state for the purpose of obtaining certain required structures with desirable properties or combination of properties suitable for the particular application. Austempered ductile iron is an alloy which has a matrix of ausferrite with no martensite or pearlite. The heat treatment of the austempering process consists of austenitising ductile iron, during which time the matrix becomes fully austenitic, quenching to the austempering temperature for a controlled time, and then cooling to a room temperature as observed in Figure 1(Keogh, 1998 and Charles, 1998).

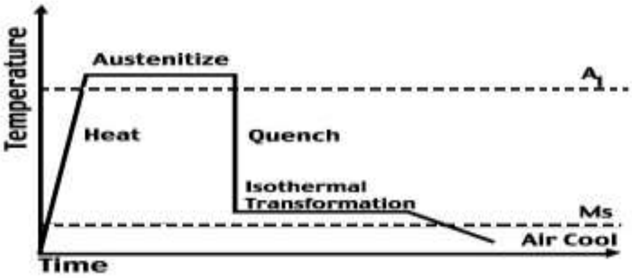

Figure 1. Schematic diagram for austempering process

During the austempering process, ADI undergoes a two-stage transformation process (Keogh, 1998 and Morrogh,1987). In the first stage, the austenite (ɣ) decomposes into bainitic ferrite (α) and carbon enriched austenite ($ɣ_{hc}$), a product known as ausferrite. This reaction can be expressed as:

$$ɣ \rightarrow α + ɣ_{hc} \quad \text{…………………………………………….I}$$

if the casting is held at the austempering temperature for too long, then carbon enriched austenite ($ɣ_{hc}$) further decomposes into ferrite (α) and carbide

$$ɣ_{hc} \rightarrow α + \text{carbide} \quad \text{………………………………….II}$$

The optimum mechanical properties are achieved with completion of the first reaction, but before the second reaction. The second reaction produces carbides, which make the material very brittle. This reaction, is therefore undesirable and must be avoided. The time period between the end of stage I and the onset of stage II is called the processing window (Keogh, 1998, and Mathew and Bolton, 2002, Rajan and Sharma, 1988).

In ductile iron, tight control of austenitising temperature is imperative if consistent properties are to be obtained. The variations in austenitising temperature shift the processing window over which the optimum properties can be attained. In general, increasing the austenitising temperature delays the austempering transformation, and shrinks the size of processing window (Fatahalla et al, 1996, Keogh, 1998).

The austenitising temperature has a significant effect on the hardenability and transformation behavior at higher temperatures, the matrix of carbon content increases and the austenite grain size is enlarged. This changes increase hardenability and reduces the rate of reaction and thus permits larger castings to be treated or less efficient quenching bath to be used (Adewuyi and Afonja, 2000, Elliott and Yazdani, 1999).

The austenitising temperature should be selected to ensure sufficient carbon transfer from the graphite nodules to the austenite matrix. The carbon solution process is both time and temperature dependent, thus high austenitising temperatures shorten the time necessary to attain a uniform carbon content in the matrix structure. However, scaling and casting distortion may result if austenitising is carried out at high temperatures (Drauglates and Boase, 1986, Fatahalla and Refaey, 2003, Rimmer, 1997).

Lower austenitising temperatures may be specified for castings which are to be machined after austempering. Lowering the austenitising temperature lowers the volume of untransformed austenite. Under the higher pressure involved in machining, the untransformed austenite transforms to martensite, thus reducing the machinability. On the other hand, a decrease in austenitising temperature frequently results in incomplete austenitision and associated problems with the microstructure and mechanical properties (Jonovak and Morton, 1984, and Fatahalla, et al, 1996).

The objective of this study is to investigate the influence of this selected austenitising temperature 850, 875, 900 and 930^{0}C and austempering time. It is believe that such an investigation will help to establish the optimum austenitising temperature. Previous studies suggest that this temperature could be within the proposed range.

II. METHODOLOGY

The ductile iron used for this investigation was produced in a medium-frequency induction furnace of 100 kg capacity. The charge consisted of 50% pig-iron ingot and 50% low-manganese steel scrap. The melt was treated with 1.8% of magnesium-ferrosilicon alloy using sandwich technique. The melt was then doped with 0.67% foundry grade of Fe-Si containing nominally 75% silicon and cast at 1400^{0}C into standard Y-block mould, which ensured a sound casting. The chemical composition of alloyed ductile iron used in this work is presented in Table 1.

Table 1. Chemical composition of treated alloy. wt-%

C	Si	Mn	S	P	Ni	Cu	Cr	Mo
3.14	2.67	0.07	0.003	0.023	0.80	0.60	0.12	0.25

Image analysis techniques were used to measure the volume fraction of ferrite, pearlite, carbides and graphite and nodule count in the as-cast structure. Specimens for the mechanical investigations were machined from the test section of the Y-block. In order to avoid segregation effect and porosity, unnotched Charpy specimens (55x10x10 mm) were machined from the bottom section of the keel block. The test specimens were initially austenitised for 2 hrs at 850, 875, 900 and 930^{0}C in a muffle furnace and then immediately austempered in a molten-salt bath at 350^{0}C for 10, 30, 60, 120, 240, 360 minutes and air-cooled to room temperature. After austempering, surface grinding was done to remove any decarburized skin that formed during heat treatment. In order to avoid the transformation of any metastable austenite to martensite, grinding speed was kept as low as possible. The austempered, unnotched, Charpy specimens were tested at room temperature. At least two samples were tested for each heat treatment condition.

For microstructural analysis, samples were taken from the impact-test specimens at positions far from the fractured area, others were taken from near the fractured area in order to determine the transformation of unstable austenite to martensite by mechanical stress. Specimens for light microscopy were prepared by a standard metallographic technique.

In order to determine the volume fraction of retained austenite, X-ray diffraction technique was carried out with Cu-Kα radiation at 40kV and 20mA. The diffractometer was equipped with chart recorder to cover the angular-2θ range from 20-120^0. The profiles were analyzed with to obtain the peak positions and the integrated intensities. The volume fraction of retained austenite (X_γ) was determined by direct comparison method using integrated intensities of the (110) and (211) peaks of ferrite, and the (111) (220) and (311) peaks of austenite. The carbon content of the austenite was determined using the relationship

$$a_\gamma = 0.3548 + 0.0044\, C_\gamma \qquad (3)$$

where a_γ is the lattice parameter of austenite in nanometer and C_γ is its carbon content (wt%). The (111) 220) and (311) peaks of austenite were used to estimate the lattice parameters.

The heat treatment of the prepared samples, after their light etching in 2% natal, improved phase identification. Heat treating involved in heating in an oven with no protective atmosphere at 260^{0}C for 5.5 hrs and cooling to room temperature. With is technique the various phases appear in different vibrant colors. These colors are: untransformed austenite-light blue, reacted high carbon austenite-light brown, ferrite-beige, eutectic carbide-white or cream, martensite-dark blue.

During point counting at last 2000 points were counted at a magnification of x500. The number of counts was increased with the decreasing volume of martensite to maintain a low standard deviation.

III. RESULTS AND DISCUSSION

3.1 Characteristics of as-cast cast iron

At observed, the as-cast microstructure of cast iron consists of graphite nodules embedded into a ferritic-pearlitic matrix as shown in Figure 2. The image analysis measurements for nodule counts, amount of ferrite, pearlte and carbide are given in Table 2.

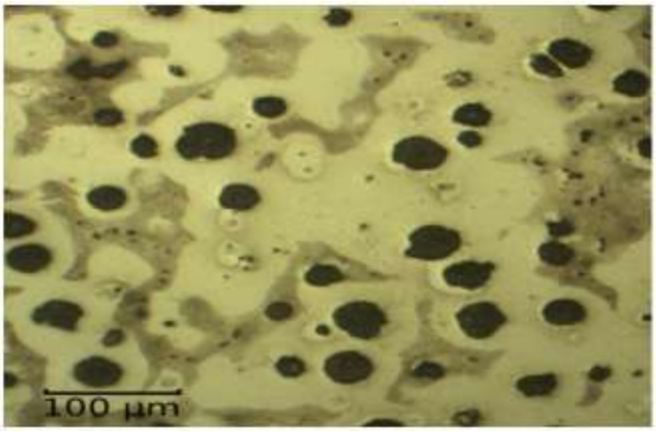

Figure 2. Initial microstructure of the as-cast cast iron with graphite nodules in black, pearlite in brown, and ferrite in white.

Table 2. Quantitative measurement of structural features of as-cast iron

Graphite vol.%	Ferrite, vol.,%	Pearlite, vol.,%	Carbide, vol.,%	Nodule count,mm^2
12.7	41.0	46.3	0.05	120

The austemperility results are presented in Figure 3 as the critical bar diameter that can be austempered without pearlite formation. As explained by Voight and Loper (1984) who have examined austemperibility, suggest the following regression function for calculating the critical bar diameter:

$$D_c = 124C_\gamma^o + 27\ (\%Si) + 22\ (\%Mn) + 16(\%Ni) + 25(\%Mo) + 1.68 \text{ x } 10^{-4}T_a^2 + 12(\%Cu)(\%Ni) + 62(\%Cu)(\%Mn) + 88(\%Ni)(\%Mo) + 11(\%Mn)(\%Cu) + 127(\%Mn)(\%Mo) - 20(\%Mn)(\%Ni) - 137 \ldots\ldots 4$$

where C_γ^o is the carbon content of the matrix austenite at the austenitizing temperature and T_a is the austempering temperature. The austenite carbon content depends on the austenitizing temperature (T_γ) and the iron composition. This dependence is normally described by the approximate expression (Dorazil, 1986).

$$C_\gamma^o = T_\gamma/420 - 0.17\ (\%Si) - 0.95 \qquad (5)$$

and the results are given in Table 3.

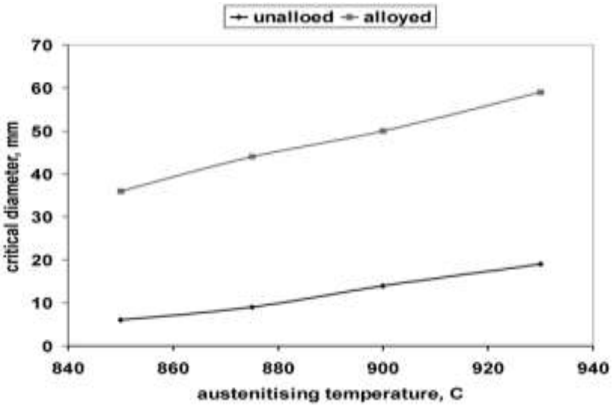

Figure 3: Variation of critical bar diameter that can be austempered without pearlite formation as function of austenitising temperature for Ni-Mo-Cu iron and unalloyed iron austempered at 350 °C (Daewish and Elliott, 1993)

The second curve in Figure 3 represents some previous measurements on unalloyed ductile cast iron austempered at 350^0C (Darwish, 1993). The critical diameters of the ductile iron increase significantly as the content of alloying additions and austenitising temperature increase.

3.2. Microstructural changes after different austenitising treatments

The morphology of the matrix after austempering depends on the austenitising temperature and austempering time. Figures 4 (a-d) show the microstructure taken from impact test specimens austenitised at 850, 875, 900 and 930^0C respectively and austempered at 350^0C for 2 hrs. as observed, all the microstructures exhibit upper bainite in the form of relatively coarse ferrite needles in an austenite matrix. The microstructure of specimen austenitised at 850^0C contains various proportions of pro-eutectoid ferrite, bainitic ferrite, and austenite (Fig. 4a). The pro-eutectoid ferrite occurs as results of low austenitising temperature, i.e. as a result of austenitising iron in the austenite-ferrite-graphite region of the phase diagram (Fig.5), Rouns et al, 1984.
As observed from Fig. 5, with increase of austenitising temperature from 850 to 930^0C the amount of retained austenite increased from 27 to 41%. The increase of austenite content with austenitising temperature can be explained by referring to the free-energy composition diagram for austenite, ferrite, and cementite phases.

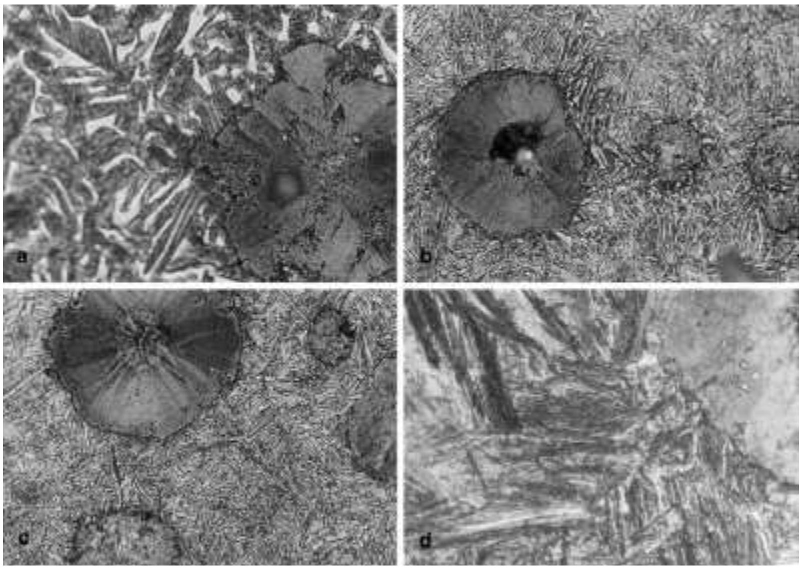

Figure. 4. Effect of austenitizing temperature on the microstructure of specimens austempered at 350^0C at 240 minutes 850^0C b) 875^0C, c) 900^0C, d) 930^0C. (Etched in 2% Nital with optical microscope).

Fig. 5b shows that as the austenitising temperature decreases from 930 to 850^0C the martensite content decreases from 33 to 19% for a 10 min austempering time. It has been shown that the rate of bainite formation increases as austenitising temperature decreases. On the other hand, the more advanced the bainite reaction the more carbon is rejected, resulting in a more stable retained austenite. Thus this austenite is less likely to transform to martensite as the specimen cools to room temperature or as stress is applied to it. Fig. 4d shows that the structure austenitised at 930^{0}C contains martensite, and as a consequence, the transformation of unstable austenite to martensite upon cooling to room temperature or the application of stress. In autempered ductile iron, it is difficult, even for the expect eye, to identify the various phases with black and white photomicrographs. To assist in the identification of the phases, heat treating technique was used. Martensite, which appeared as dark blue was evident. This accounts for the low-impact energy results as observed in Fig.6.

Another important point is that with the austenitising temperature more carbon is taken into solution by the original austenite as observed in Table 3. Thus, the austenite remaining after austempering may be stabilized even if a small percentage of ferrite is formed. Also as observed in Table 3, increasing the austenitising temperature from 850 to 930^{0}C increases the carbon content at the austenitising temperature from 0.63 to 0.81%. The actual carbon content of the austenite was made using X-ray diffraction techniques (Fig. 5c)

Table 3. Matrix austenite carbon content after austenitisation

Austenitising temperature, ^{0}C	850	875	900	930
Carbon content in austenite, vol.%	0.63	0.68	0.74	0.81

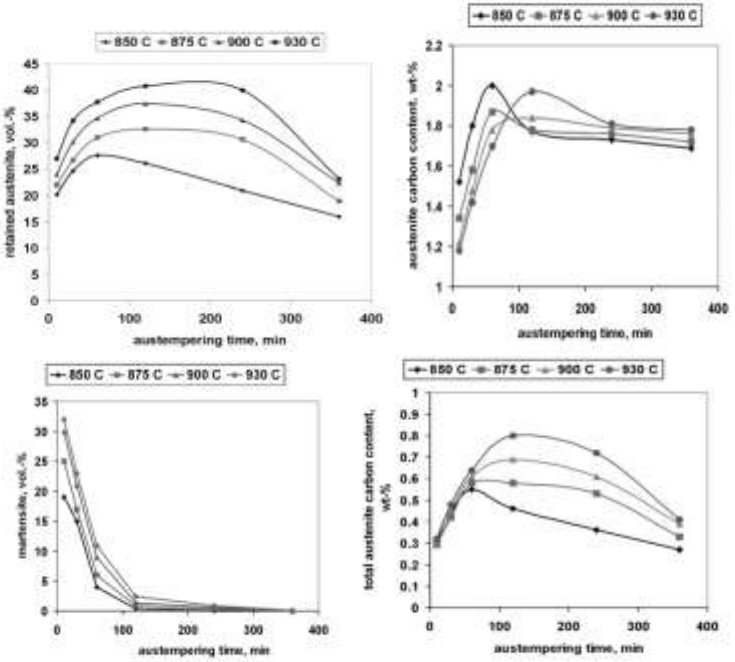

Figure 5: Variation in a) volume of retained austenite; b) martensite; c) austenite carbon content; d) total austenite carbon content; with austempering time for iron austempered at 350 °C and austenitised at 850, 875, 900 and 930 °C for 2 hrs

3.3. Kinetics of transformation

The kinetics of the process can be revealed by the structural parameters, austenite volume fraction (X_γ) and austenite-carbon content (C_γ), both determined by X-ray diffraction technique. The variation of X_γ, and (C_γ versus austempering time for the heat treated ductile iron are shown, respectively in Figs.5 (a and c). it is observed that a high austenitising temperature of 930^{0}C results in a relatively larger volume of retained austenite when compared to the iron austenitised at lower temperatures. However, this iron has the lowest austenite-carbon contents for all the austempering times. In contrast, the iron austenitised at 850^{0}C contains lower levels of retained austenite but the highest carbon contents in the retained austenite. This austenite is thermally and mechanically stable which leads to optimum impact properties as observed in Fig. 6.

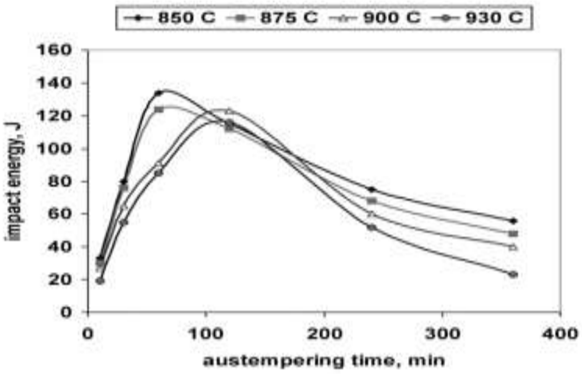

Figure 6: Variation of impact energy with austempering time after austenitising at 850, 875, 900 and 930 °C for 2hrs

In Fig. 5d the total austenite-carbon contents in the original austenite matrix ($X_γC_γ$) are plotted versus austempering time. The figure shows that the magnitude of the product $X_γC_γ$ for an austenitising temperature of 930^0C increases to a higher maximum value than for the sample treated at 850^0C. Therefore, less original austenite carbon is dissolved within the austenite matrix at the lower austenitising temperature.

Increasing the austenitising temperature to 930^0C increases the austempering time needed to reach the maximum retained austenite. at 930^0C, the maximum is reached for about 2 hrs, but at 850^0C the maximum is reached for less than 1 hr. this is in agreement with the theory, and is explained by the free-energy diagram.

The austempering time has an important influence on the mechanical properties and microstructure. As the austempering time is extended to 2 hrs, the volume of retained austenite starts to decrease. This decrease is as the result of second –stage reaction in which the high carbon starts to decompose into ferrite plus carbides. As observed in Fig. 5e, the austenite-carbon content is also lowered and leads to further transformation to bainitic ferric and carbides. The precipitation of carbides reduces the impact-energy values as observed in Fig.6

IV. CONCLUSIONS

The following conclusions could be drawn from this research work:

1. Decreasing the austenitising temperature reduces the volume of retained austenite, but increases the austenite-carbon content and optimized the impact toughness.
2. The critical diameter of ductile iron increases with increase of austenitising temperature and alloying addition.
3. Heat treatment at 850^0C resulted in pro-eutectoid ferrite in the microstructure. This is attributed in lowering austenitising temperature which reduces the stability of carbon in austenite and introduces a three-phase region of ferrite, austenite, and graphite.
4. The drop in impact energy at the austenitising temperature of 930^0C appeared to be caused by a reduction in austenite-carbon content as a result of carbides precipitation

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