Stepped Austempering of GGG 40 Ductile Cast Iron

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The effect of stepped austempering heat treatment (900°C, 60 min; 380°C, 15–120 min; 320°C, 60–360 min) on the microstructural and mechanical properties of an unalloyed ductile iron having a chemical composition of 3.86% C, 2.1% Si, 0.4% Mn, 0.042% Mg, 0.059% S and 0.07% P was studied. These properties were compared with those obtained by single austempering heat treatment (900°C, 60 min; 380°C, 60–360 min). Microstructure, hardness, Charpy impact V-notch and tensile properties were evaluated after each heat treatment cycle. The average austenite carbon content and retained austenite content have been evaluated by X-ray diffraction. The fractography of tensile heat treated specimens was examined and evaluated using SEM. The results obtained showed that a stepped austempering treatment could be used to increase the strength without significant loss in ductility or impact energy compared with single austempering. The optimum combination of mechanical properties is obtained at first step austempering time of 45 min. For the other conditions, The UTS increases slightly by increasing the second step austempering time. The average austenite carbon content results indicate that stage II begins after 120 min of the second step austempering time which leads to reduction in ductility and impact toughness as a result of carbides precipitation. Decreasing the first austempering time, accelerates the stage I reaction in the second step treatment but causes a loss in the strength and hardness and an increase in ductility and impact energy. Analysis of fractured surfaces reveals that the fracture of stepped austempered ductile cast iron is a ductile fracture and this mode almost remains unchanged with different conditions.

KEY WORDS: ductile cast iron, austempering, retained austenite, mechanical properties, bainite, X-ray diffraction.

1. Introduction

Several studies1–3) have shown that austempering can be used to enhance the mechanical properties of ductile iron so that they are comparable of low-alloyed steel. Bainitic ductile iron is more and more applied in high stress machine parts such as gears, crankshafts, mining car wheels, chain links, connecting rods, transportation and storage casks for radioactive materials.4) Bainitic transformation is accomplished into two stages, I and II. At lower austempering temperatures, which are used to produce the high strength grades of austempered ductile iron, the stage I reaction transforms the low carbon austenite resulting from austenitizing into bainitic ferrite, carbide and high carbon austenite. At higher austempering temperatures, which are used to achieve the high ductility grades of austempered ductile iron (ADI), the stage I reaction transforms the low carbon austenite into bainitic ferrite and high carbon austenite. In stage II reaction the high carbon austenite breaks down into ferrite and carbides. The aim of the present study is to study the effect of the two stepped austempering isothermal treatment and time on the resulting microstructure and mechanical properties of ADI using Tensile testing, Charpy impact V-notch test, X-ray diffraction and scanning electron microscope (SEM) and to analyze the obtained results to determine the optimum heat treatment process, and identify its optimum parameters which achieve the most favorable combination of mechanical properties.

2. Experimental Work

The chemical compositions of the ductile iron used is 3.86% C, 2.1% Si, 0.4% Mn, 0.042% Mg, 0.059% S and 0.07% P. This ductile iron is GGG 40 (DIN 1693) was provided by El-Nasr Casting Company in Egypt, and it is being used for producing tubes by centrifugal casting. The average mechanical properties of as-received material was examined and found to be as follows: UTS, 420 MPa; yield strength, 300 MPa; Elongation, 16%; Young’s modulus, (15–17 · 10^3); average hardness 175 BHN; and 15.5 J/cm^2 impact toughness. For each heat treatment cycle, three different types of test specimens were machined from the as-received material. Samples (20×20×5 mm) were prepared to follow the microstructural changes and the retained austenite content and its carbon content during the second step austempering after a first one at 380°C. Tensile test specimens were machined on 5 mm gauge diameter standard specimens according to DIN 50/25, as shown in Fig. 1. The standard Charpy V-notched impact specimens were fabricated. The as received annealed ferritic specimens were heat treated to change the microstructure of their steel-like matrix. Heat treatment was carried out using neutral salt baths to avoid oxidation, carburizing and decarburizing. All specimens were austenitized at 900°C for 60 min
in a salt bath of type GS-540 (80% BaCl₂, 20% NaCl). To avoid cracking and warping which might result from rapid heating of the specimens, they were initially heated to 400°C inside an air furnace for 20 min before being immersed into the salt bath at 900°C. To eliminate errors related to the austenitizing temperature, specimens were completely immersed inside the bath to the same level and the temperature of the bath was checked and calibrated to an accuracy ±5°C. Austempering was achieved by rapidly transferring specimens from the austenitizing furnace to a salt bath AS-140 (100% Na₂NO₃) held at the first austempering temperature 380°C for different periods of time (15–120 min) followed by transfer to a second salt bath at 320°C for times range from 60 to 360 min. Quenching was achieved by rapid cooling of the austenitized specimens in oil to room temperature. The heat treatment cycles are shown schematically in Fig. 2. Microscopic examination was used to study microstructure changes resulting from different heat treatment processes. Changes in retained austenite content and its carbon content were measured for polished specimens by X-ray diffraction using monochromatic Cu Kα radiation with a Ni filter, at 30 kV and 20 mA. An XRD-3A Shemadzu computerized machine was used to scan the angular 2θ ranges from 40 to 85°. The carbon content of retained austenite was calculated from the angular position of the FCC austenite peaks and the volume fraction of retained austenite was determined using two different techniques, one is the direct comparison method and the other is Miller’s technique. Both techniques are assuming that the austenite and the ferrite are the only matrix phases present in the specimen. Methods of X-ray calculations are given in the appendix. Charpy V-notch impact test was carried out at room temperature and three specimens were tested for each cycle and the average was taken. A 10 kN universal testing machine was used for tensile test with cross head speed 10 mm/min; two specimens were tested for each heat treatment condition. The fracture surface was examined using SEM. The heat treated conditions will be named as group A, B, C, D, E and F according to the first step austempering time which is 15, 30, 45, 60, 90 and 120 min respectively. This naming is to facilitate discussion. The mechanical properties obtained was compared with that obtained from single austempering at 380°C.

3. Results and Discussion

3.1. Microstructure and X-ray Diffraction

Microstructure of the as-received material was examined and found to be consisted of graphite nodules embedded in a ferritic matrix. Figure 3 shows a typical structure of ferritic ductile iron, due to the ferritization annealing process that had been performed after casting the tubes, as reported by the manufacture. It has high uniformity of graphite nodule distribution and a high number of nodules per unit area (1200–1500 nodules/mm²). In the present study, the stepped heat-treated specimens were studied by the optical microscope. X-ray diffraction analysis was carried out to ascertain the amount of retained austenite and its carbon content. The austenitizing temperature, first step austempering temperature and time determine the structural features of the iron and the average austenitic carbon content before the second step austempering treatment and consequently play a significant role in determining the improvement in properties and time taken to achieve the improvement during the second austempering treatment. The aim of the second step treatment is to continue the stage I reaction without a simultaneous stage II reaction in the eutectic cell. This is achieved by decreasing the austempering temperature which accelerates the stage I reaction without a simultaneous stage II reaction in the eutectic cell. The second step temperature was 320°C and the effect of this temperature is discussed below. The second step reaction is the transformation of austenite to bainitic ferrite, carbide and high carbon austenite. The austenite matrix that can transform is not of uniform carbon content. So, the transformation will start in areas of lower carbon content in the intercellular areas and in the center of blocky austenite areas. Figure 4 shows typical microstructures after a first step treatment of 45 min at 380°C and step at 320°C for 60, 120, 360 min respectively. There is an evidence of mixed (lower and upper)
bainitic ferrite plates and some areas of blocky austenite. It is difficult to distinguish the structural variation in the three conditions of different second step austempering time as shown in Fig. 4. It was not possible to measure the amount of retained austenite quite accurately by optical methods. More exact and reliable determination of retained austenite was obtained by X-ray diffraction. Typical spectrum for condition (900°C, 60 min; 380°C/45 min; 320°C/60, 120, 360 min) is shown in Fig. 5. In this range three diffraction peaks of ferrite phase namely (110), (200), (211) and four diffraction peaks of austenite phase namely (111), (200), (220) and (311) can be distinguished. Diffraction peaks (200) of austenite and (211) of ferrite phase were only considered in calculation by direct comparison method\textsuperscript{5)} while peaks of (220) and (311) for austenite and (211) of ferrite are only considered in calculation by Miller’s method.\textsuperscript{6)} The retained austenite content and the average austenite carbon content were measured for ductile irons cooled to room temperature from the second step austempering temperature. The results of these parameters are given in Tables 1 and 2 respectively. The values listed in Table 1 are the average of direct comparison and Miller’s methods. The variation of austenite volume fraction with second step austemper-

![Fig. 4. Microstructure of group C stepped austempered specimens.](image)

![Fig. 5. X-ray diffraction spectrum obtained by specimen (900°C, 60 min; 380°C/45 min; 320°C/60 min.).](image)

| Time, min. | Group A | Group C | Group D | Group F |
|-----------|---------|---------|---------|---------|
| 0         | 43      | 40      | 16      | 32      |
| 60        | 27      | 23      | 16.5    | 14.8    |
| 120       | 20.5    | 19.8    | 14      | 13.8    |
| 360       | 18.2    | 13.7    | 12.4    | 12.1    |

Table 1. Percentage of retained austenite during second step austempering.
pering time is shown in Fig. 6. The retained austenite content is found to decrease with increasing second step austempering time at different conditions (A, C, D and F). Figure 6 shows also that increasing the first step time causes more reduction in the retained austenite content. When any austenite transforms in the second step treatment there will be an increase in the local austenite carbon content. Unreacted austenite of a high carbon content, that is formed after the first treatment, will transform to higher carbon austenite and ferritic bainite representing a decrease in the measured retained austenite during the second step austempering. The pattern of change in Fig. 6 is in consistent with this explanation. A number of methods may be used to analyze the carbon content in austenite in cast irons8): a) microprobe analysis, b) measurements of lattice parameters of austenite by X-ray diffraction, c) measurements of the hardness of martensite, d) chemical analysis.

X-ray diffraction was adopted in this study. This was achieved by determining the position of the available diffraction peaks and converting the diffracted angles using standard techniques for calculating the lattice parameter of austenite \(a\) (nm). The carbon content of the austenite \(C\) (mass%) was then computed using the relationship9): \(C = \frac{|a - 0.3548|}{0.0044}\). The variation of austenite carbon content during conditions (A, C, D and F) is given in Fig. 7. The austenite carbon content increases continuously to a maximum at 120 min second step austempering time, and then decreases owing to the stage II reaction in which high carbon austenite transforms to ferrite and carbides. Bayati and Elliot obtained similar results.\(^{10}\) But in their steel, the decrease of carbon content occurs at later times due to the effect of alloying elements (Mo and Cu). Compared with single austempering, retained austenite resulting from stepped austempering has higher thermodynamic and mechanical stability than that of single austempering owing to the higher carbon content in it.

Table 2. Percentage of average austenite carbon content during second step austempering, mass%.

| Time, min. | Group A | Group C | Group D | Group F |
|-----------|---------|---------|---------|---------|
| 0         | 1.26    | 1.52    | 1.53    | 1.43    |
| 60        | 1.77    | 1.78    | 1.87    | 1.77    |
| 120       | 1.96    | 1.96    | 1.96    | 1.83    |
| 360       | 1.67    | 1.75    | 1.65    | 1.61    |

3.2. Mechanical Properties

Figure 8 shows the relation between the matrix hardness and the austempering time interval for the second step austempering. The curves show that the value of the matrix hardness increases slightly with time owing to the decrease of retained austenite\% by increasing second austempering time. It can be seen that as the first step time increases, the hardness values increases. Group C (900°C; 60 min; 380°C/45 min; 320°C/60, 120, 360 min) shows the maximum hardness values over all time intervals. Although this group has a considerable great amount of retained austenite for which a lower hardness would be expected. On the contrary, it has the highest hardness. This may be attributed to another factor that influencing the hardness. This factor is the nature of the second step austempering product. These specimens have an average retained austenite of 40\% just before quenching to the second step temperature. This implies that large proportion of this retained austenite will transform into lower bainite, which is harder than upper bainite, and it is the responsible for the noticed increase in hardness. Compared with single austempering at 380°C, Fig. 8 indicates that stepped austempering shows a higher hardness than single austempering overall second step time, which ensures that a large proportion of lower bainite is present in the case of stepped austempering. At low austempering temperatures in the lower bainite range, a high strength is produced by single austempering. Features of microstruc-
ture that promote strength are fine structure of the ferrite needles, dispersed carbides and low levels of retained austenite. This lower bainite morphology and low levels of retained austenite contribute to the low ductility obtained at low austempering temperatures. The aim of stepped austempering is to obtain higher levels of strength similar to that obtained at single-low austempering temperatures but with higher levels of elongation and notched impact energy than those obtained in single-low austempering temperatures. Measurements of impact energy as a function of the second step austempering time are shown in Fig. 9. There is a slight decrease in the impact energy as the second step austempering time increases up to two hours. It can be seen that, at any austempering time, the shorter the first step austempering time the higher the impact energy obtained. It appears that the best results have been obtained with group A (900°C, 60 min; 380°C/15 min; 320°C/60, 120, 360 min), which has the maximum retained austenite overall periods of second step austempering. The single austempering treatments at 380°C, which has a considerable high impact energy is sharply reduced due to the start of stage II reaction in which carbon enriched austenite transforms to ferrite plus carbides. On the contrary, the reductions in impact energy after 2 h for stepped austempering treatments are less than that of single austempering at 380°C. Nazar Boland and Elliot got similar results for alloyed ductile iron but with longer time intervals due to the shift of The C-curve to the right hand side as a result of alloying elements addition. The variation of UTS with second step austempering time is shown in Fig. 10. It is clear that all stepped austempering treatments result in greater UTS than single austempering treatment at 380°C. Group C (900°C, 60 min; 380°C/45 min; 320°C/60, 120, 360 min) shows the highest strength. This may be attributed to the presence of large proportion of lower bainite, which is harder than upper bainite. Figure 11 shows the variation of ductility (elongation%) with second step austempering time. It shows that as the second austempering time increases, the elongation% decreases. It can be observed that elongation% values after 360 min drop to about 50% of its values at 60 min second step austempering time, which is mainly due to stage II reaction and the formation of carbides. Carbides are known to decrease the elongation% of ductile iron but don’t affect the strength. Group C (900°C, 60 min; 380°C/45 min; 320°C/60, 120, 360 min) has a reasonable ductility but lower than group A (900°C, 60 min; 380°C/15 min; 320°C/60, 120, 360 min) and group B (900°C, 60 min; 380°C/30 min; 320°C/60, 120, 360 min), which have greater amount of retained austenite. It can be seen that single austempering at 380°C has higher ductility than stepped austempering up to 280 min but when the time increases over 280 min the elongation are very close to each other that may be attributed to the precipitation of carbides. The improvement in mechanical properties especially UTS during the stepped austempering is due to the lower bainite morphology and the increase in the austenite carbon content, which will improve the mechanical stability of the austenite. The mechanical properties obtained indicate that with the present iron there is only a slight difference in the austempered properties and the time to achieve these properties. The fractured surface of as-received annealed material in tension condition is shown in Fig. 12. The ductile rupture mode was obtained; the fracture surface is seen to consist of widely spaced large dimples, which are related to the graphite nodules, separated by a region with arrays of fine dimples. Graphite is present as spherical nodules, which leads to the large dimples with intensive local strain of the matrix. Figure 13 shows the tensile fracture surface of the conditions (900°C, 60 min; 380°C/15 min; 320°C/360 min), (900°C, 60 min; 380°C/30 min; 320°C/360 min), (900°C, 60 min; 380°C/45 min; 320°C/360 min), and
(900°C, 60 min; 380°C/45 min; 320°C/120 min). This figure illustrates the effective appearance of a dimple ductile fracture surface. It is observed that the mode of the fracture almost remains without change at different austempering conditions. No indication of cleavage fracture was obtained and the micrographs include dimples formed by micronecking. The large microvoids shown in the figure are due to the presence of spheroidal carbide particles. These microvoids are initiated at the interface between the matrix and the carbide particles and also at imperfections such as microporosity and microcracks. Void formation and volumetric expansions of ductile iron, which are plastically deformed in simple tension, have been widely reported in literature.14)

4. Conclusions

Measurements of the austempering kinetics and mechanical properties during stepped austempering heat treatment for the present ductile iron leading to the following results:

(1) Microstructure after stepped austempering heat treatment consists of mixed (lower and upper) bainitic ferrite plates, graphite nodules and some areas of austenite. It was difficult to distinguish the structural variation in the conditions with the same first step austempering time and different second austempering times.

(2) Measurements of retained austenite content for different cycles indicate that the retained austenite content decreases with longer second step austempering time. The carbon concentration of retained austenite for specimens transformed indicates that precipitation of carbides begins after 120 min second step austempering time, which results in a reduction of mechanical properties mainly elongation and notched charpy impact energy. It also shows that stage I reaction continues up to 120 min second step austempering time.

(3) A stepped austempering treatment condition (900°C, 60 min; 380°C/45 min; 320°C/60, 120, 360 min) gives the optimum combination of mechanical properties. The UTS slightly increases with longer second step time. Compared with single austempering at 380°C, it shows higher UTS without significant loss in elongation and notched impact energy.

(4) Fractography shows that the fracture surface of the austempered ductile cast iron is a ductile mode of fracture and this mode almost remains unchanged for different heat treatment conditions. The major drawback of the stepped austempering treatment is the cost implication of double austempering treatments.

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Appendix. X-ray Diffraction Calculations

The amount of retained austenite and the carbon content of austenite were measured by X-ray diffraction (XRD) techniques, using monochromated Cu Kα radiation at 30 kV and 20 mA on the polished specimen. The volume fraction of retained austenite was calculated using two techniques.

The first one is Miller’s technique\(^6\):

\[
X_\gamma = \frac{1.41 \gamma}{(1\alpha + 1.41 \gamma)}
\]

where \(I\gamma\) is the average of the integrated intensity from (220)\(\gamma\) and (311)\(\gamma\) planes.

And \(1\alpha=1\alpha(211)\).

Where \(I\gamma(hkl)\) and \(I\alpha(hkl)\) denotes the integrated intensity (area under the peaks of the hkl reflection from the respective phase).

The second technique is direct comparison method\(^5\); this method is of greatest metallurgical interest because it can be applied directly to polycrystalline specimens. The volume fraction estimation can be made from measurements of the integrated intensities of the BCC ferrite and FCC austenite phases assuming they are the only matrix phases present. The ratios of intensities of diffraction peaks from these phases is given by:

\[
I\gamma(hkl)/I\alpha(hkl) = R\gamma(hkl)/R\alpha(hkl) * X_\gamma/X_\alpha
\]

where:

\(I\gamma(hkl)\) and \(I\alpha(hkl)\): integrated intensity from a given \((hkl)\) from \(\gamma\) and \(\alpha\) phase respectively.

\(X_\gamma\) and \(X_\alpha\): volume fraction of \(\gamma\) and \(\alpha\) respectively.

And the constants \(R\gamma(hkl)\) and \(R\alpha(hkl)\) are given by the expression:

\[
R = \frac{1}{V^2[F^2P\Lambda p]e^{-2m}}
\]

Where: \(V\)=atomic volume of unit cell, \(F\)=structure factor, \(P\)=multiplicity factor, \(\Lambda p\)=Lorenz-polarization factor and \(e^{-2m}\)=temperature factor.

The R factors have been calculated for the (200) peak for \(\gamma\) iron and the (211) peak for the \(\alpha\) iron.