Characterization of Austempered Ferritic Ductile Iron

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Abstract. The ductile iron (DI) has graphite nodules enclose in ferrite envelop in pearlitic matrix. The pearlitic matrix in DI was converted to ferritic matrix through heat treatment. This heat treatment includes austenitization of DI at 900°C for 1h, followed by furnace cooling to 750°C & hold for 1h, then again furnace cooling to 690°C hold for 2h, then samples were allowed to cool in furnace. The new heat treated DI has graphite nodules in ferritic matrix and called as ferritic ductile iron (FDI). Both DIs were austenitized at 900°C for 1h and then quenched into salt bath at 325°C. The samples were soaked in salt bath for 60, 120, 180, 240 and 300 min followed by air cooling. The austempered samples were characterized with help of optical microscopy, SEM and X-ray diffraction analysis. Austempering of ferritic ductile iron resulted in finer ausferrite matrix as compared to ADI. Area fraction of graphite, ferrite and austenite were determining using AXIOVISION-SE64 software. Area fraction of graphite was more in FDI than that of as cast DI. The area fraction of graphite remains unaffected due to austempering heat treatment. Ausferritic matrix coarsened (feathered) with increasing in austempering time for both DI and FDI. Bulk hardness test was carried on Rockwell Hardness Tester with load of 150 kgf and diamond indenter. Hardness obtained in as cast DI is 28 HRC which decreased to 6 HRC in FDI due conversion of pearlitic matrix to ferritic matrix. Hardness is improved by austempering process.

1. Introduction

Ductile Iron (DI) had carbon in from of graphite like in a gray cast iron but in nodular form which gives it superior mechanical property like tensile strength and toughness. Graphite flaks in a gray cast iron act as stress contraction where as graphite nodules act as crack arresters in DI[1]. The strength of DI is also depends upon it matrix. Pearlitic matrix had high strength, good wear resistance with fair amount of ductility. Ferritic matrix had good ductility with tensile strength equivalent to that of low carbon steel. Ferritic - pearlitic matrix had in-between properties that between ferritic and pearlitic DI[2][3]. The microstructure of matrix can be obtained in as cast condition or by heat treatment of casting[4].

Austempered ductile iron (ADI) is new member in cast iron family. It had very good combination of mechanical properties like tensile strength, wear resistance, toughness and ductility at low cost[5]. Due to this ADI used in many engineering application like automobiles parts (gear, cam shaft, etc.).[6]. The strength of ADI is due to it Ausferritic matrix. Ausferrite is mixture of acicular ferrite and carbon stabilized austenite[7]. The ausferrite content in matrix of ADI depends upon the austempering heat treatment parameter, chemical composition and initial (prior to the austempering) microstructure of DI[8][9]. The austempering reaction takes place in two stages, in first stage austenite gets converted into acicular ferrite and carbon stabilized austenite (CSA) and in second stage CSA is converted into acicular ferrite and carbides[10].
The DI usually had ferritic pearlitic matrix with graphite nodules dispersed in microstructure. The austempering of DI doesn’t affect the area fraction of graphite[11]. Thus it is clear that the matrix and chemical composition of DI plays important role in determining the ausferritic content in ADI and therefore the mechanical properties of ADI.

In this work, two DI with different microstructure one with pearlitic ferritic matrix and other one with ferritic matrix were selected. The ferritic matrix was obtained by heat treatment of pearlitic - ferritic DI. The both DI have same chemical composition and same austempering heat treatment parameter. Effect of austempering process on initial matrix content of DI is studied with help characterization techniques like optical microscope (OM), scanning electron microscope (SEM), X-ray diffraction (XRD) and hardness.

2. Experimental Details

The ductile iron was produced in a induction furnace with capacity of 150kg. Samples were cast in CO2 mould. The cast sample dimensions diameter 20 mm and length 300 mm. The chemical composition is given in table 1.

| Elements      | Weight (%) |
|---------------|------------|
| Carbon        | 3.36       |
| Silicon       | 2.95       |
| Copper        | 0.59       |
| Nickel        | 0.76       |
| Manganese     | 0.40       |
| Iron (balanced) | 91.93     |
| Carbon Equivalent (CE) | 4.34      |

Table 1. Chemical composition

The ductile iron was heat treated to convert pearlitic matrix into ferritic matrix. The schematic of heat treatment was shown in figure 1. The heat treatment includes austenitization of DI at 900°C for 1h, followed by furnace cooling to 750°C & hold for 1h, then again furnace cooling to 690°C hold for 2h, then samples were allowed to cool in furnace. The new heat treated DI has graphite nodules in ferritic matrix and called as ferritic ductile iron (FDI).

In the first batch of austempering, FDI or heat treated ductile iron were austenitized at 900°C for 1h and then quenched into salt bath at 325°C. The samples were soaked in salt bath for 60, 120, 180, 240
and 300 min followed by air cooling. In the second batch of austempering DI samples were austempered with same parameters. The figure 2 shows schematic of austempering process for both irons. The heat treatment matrix and samples codes were given in table 2.

| S. No. | Austenitization temperature (°C) & Time (h) | Austempering temperature (°C) | Austempering Time (h) | Code |
|-------|--------------------------------------|-----------------------------|----------------------|------|
| 1     | Ferritic Ductile Iron                |                             |                      | A0   |
| 2     | 900°C & 1h                           | 325°C                       | 1                    | A1   |
| 3     | 900°C & 1h                           | 325°C                       | 2                    | A2   |
| 4     | 900°C & 1h                           | 325°C                       | 3                    | A3   |
| 5     | 900°C & 1h                           | 325°C                       | 4                    | A4   |
| 6     | 900°C & 1h                           | 325°C                       | 5                    | A5   |

| 7     | Ductile Iron                         |                             |                      | B0   |
| 8     | 900°C & 1h                           | 325°C                       | 1                    | B1   |
| 9     | 900°C & 1h                           | 325°C                       | 2                    | B2   |
| 10    | 900°C & 1h                           | 325°C                       | 3                    | B3   |
| 11    | 900°C & 1h                           | 325°C                       | 4                    | B4   |
| 12    | 900°C & 1h                           | 325°C                       | 5                    | B5   |

The heat treated and austempered samples were characterized with help of optical microscopy (OM), scanning electron microscope (SEM) and X-ray diffraction analysis (XRD). The cross sections of samples were fine polished up to 4/0 emery paper followed by polishing over soft velvet fiber cloth using suspended alumina particles in water. Samples surface were cleaned off to remove any dust, foreign material. Samples were etched in Nital (97ml C2H5OH, 3ml nitric acid) prior to characterization. The optical microstructure captured using CARL ZEISS type (PL–A 662, Axioskop2 MAT) microscope.

The XRD analysis of samples were carried on Philips X’Pert PRO PANalytical using Cu Kα target and wavelength $\lambda = 1.5406$ Å. The samples were analyzed over range of $\theta$ between $30 – 110^\circ$. Austenite lattice parameter ($a_\gamma$), percentage of carbon content in austenite ($C_\gamma$)[12] and ferritic cell size ($d$) were obtained from XRD analysis; details of this procedure were reported elsewhere[13][14].

The image analysis was carried on using AXIOVISION-SE64 software, version REL 4.9.1. The figure 3 (a) shows image of as cast ductile iron. After processing through software various phases indentified with annotation image in this case it is graphite shown in figure 3 (b). The area covered is measured by software and that’s gives area fraction of that phase. Similarly, 8 images at 200X magnification at different location were processed and average value is reported.
Hardness was measured on Rockwell hardness tester. Rockwell ‘C’ scale was used. For ‘C’ scale 120° diamond indicator with load of 150kgf was selected. Average of five hardness reading is reported.

3. Results and Discussions

3.1. Microstructure

As cast microstructure of ductile iron is shown in figure 4. The figure shows graphite nodules embedded in ferrite surrounded by pearlite matrix hence is called as pearlitic ductile iron. The figure 5 shows optical microstructure of heat treated ductile iron; it shows graphite nodules surrounded by ferrite matrix and some small patched of pearlite. Due to heat treatment pearlite matrix gets converted into ferrite and graphite.

The SEM images of austempered ductile iron and ferritic ductile iron (FDI) are shown in figure from 6 to 15. The figure 6 and 7 shows SEM images of austempered FDI and ADI for 1h of austempering. Both figure shows graphite nodules surrounded by ausferrite matrix and martensitic laths.

When samples were quenched from Austenitization temperature to austempering temperature ferrite gets nucleated at austenite grain boundary and austenite graphite interface. As the ferrite can no longer hold excess carbon in matrix it rejects carbon into surrounding austenite. Thus austenite becomes more stable and doesn’t transform into martensite. The untransformed austenite is called as carbon stabilized austenite (CSA). The amount of CSA formed is depends upon the austempering time. As austempering
time is less some austenite doesn’t saturate with carbon atoms and get converted into martensite. Hence after 1h of austempering same martensite is observed.

The figure 8 and 9 shows SEM images of austempered FDI and ADI after 2h of austempering shows ausferritic matrix and graphite nodules. Austenite gets saturated with carbon atoms after 2h and doesn’t convert into martensite; hence no martensite is observed after air cooling. After 3h of austempering some coarsening of ausferritic matrix is observed in both austempered samples (figure 10 & 11).

Further coarsening of ausferritic matrix was observed after 4h and 5h of austempering. Figure 12 and 13 shows coarse ausferrite matrix and graphite nodules after 4h of austempering of FDI and DI respectively. Similarly figure 14 and 15 shows SEM images after 5h of austempering for FDI and DI respectively. After 5hr of austempering decomposition of ausferrite into ferrite and carbon was observed in case of ductile iron figure 15.
3.2. Image Analysis

As cast ductile iron had 13% area fraction of graphite, 75% area fraction of pearlite and 12% area fraction of ferrite. After heat treatment of ductile iron, ferritic ductile iron had 20% area fraction of graphite, 70% area fraction of ferrite and 10% area fraction of pearlite. As a result of this heat-treatment pearlitic matrix in ductile iron gets converted into ferritic matrix, due to the slow cooling rate/ furnace cooling. Excess carbon presents in the pearlitic matrix get accommodated in the graphite nodule. Due to this the area fraction of graphite increases from 13% (DI) to 20% and area fraction of ferrite increases from 12% (DI) to 70% in FDI.

The results of images analysis of austempered samples is given in table 3. After austempering graphite area fraction remains unchanged in both DIs. In case of FDI area fraction of graphite various 19 – 20% and in case of DI it various from 11 to 13%. Thus the graphite area fraction remains unaffected by austempering process.
Table 3. Image Analysis (Area Fraction in %)

| Austempering Time (h) | Ferritic Ductile Iron | Ductile Iron |
|-----------------------|-----------------------|-------------|
|                       | Graphite | CSA | Ferrite | Graphite | CSA | Ferrite |
| 1                     | 19       | 32  | 28      | 12       | 28  | 38      |
| 2                     | 20       | 46  | 31      | 13       | 39  | 42      |
| 3                     | 19       | 31  | 36      | 11       | 31  | 46      |
| 4                     | 19       | 27  | 46      | 13       | 31  | 41      |
| 5                     | 20       | 26  | 49      | 13       | 30  | 57      |

The area fraction carbon stabilized austenite increases initially up to 2h and then decreases with austempering time. Maximum area fraction of CSA is obtained at A2 (46%). The area fraction of ferrite increases with austempering time. Maximum area fraction of ferrite is 57% at B5. The area fraction of CSA initially increases due to first stage of austempering process. Area fraction of ferrite increases due to results of first and second stage of austempering process.

3.3 XRD Analysis

The results of XRD analysis is given in table 4. Ferrite cell size represents ferrite grain size. In both DIs, ferrite cell size increases with austempering time. As the austempering time increases more carbon atoms from ferrite diffused out in to surrounding austenite and ferrite grows. This indicates coarsening of ausferritic matrix.

In case of austempering of FDI the ferrite cell size is lower than that of austempered ductile iron. From the image analysis, it is clear that the area fraction of graphite is not affected by austempering process. Thus it is clear that carbon which is in graphite form will not play any role in austempering process. The initial carbon content (before austempering process) of the FDI matrix is less as compared to that of ductile iron, because ferrite can hold only 0.0025wt% of carbon. So at the austenitization temperature austenite carbon content is less in case of FDI than that of DI. Upon quenching at austempering temperature, lesser carbon content of austenite promotes more ferrite nucleation thus resulted in finer ferrite cell size in austempered FDI.

Table 4. XRD Analysis Results

| Austempering Time (h) | Ferritic Ductile Iron | Ductile Iron |
|-----------------------|-----------------------|-------------|
|                       | Ferrite Cell Size | Austenite Lattice Parameter | Carbon Content of Austenite | Ferrite Cell Size | Austenite Lattice Parameter | Carbon Content of Austenite |
|                       | nm   | nm         | Wt %   | nm   | nm         | Wt %   |
| 1                     | 16.0 | 0.3621     | 1.35   | 19.9 | 0.3621     | 1.36   |
| 2                     | 18.5 | 0.3621     | 1.50   | 23.9 | 0.3617     | 1.41   |
| 3                     | 20.6 | 0.3615     | 1.52   | 25.2 | 0.3620     | 1.59   |
| 4                     | 23.7 | 0.3627     | 1.64   | 27.8 | 0.3624     | 1.57   |
| 5                     | 24.5 | 0.3628     | 1.66   | 27.3 | 0.3623     | 1.50   |

As the austempering time increases austenite gets saturated with carbon atom and hence lattice parameter of austenite increases thus the increase carbon content of austenite. As the rate of nucleation of ferrite is more in FDI thus rate of first stage of austempering reaction. As the rate of austempering reaction is more in austempering of FDI, after 5h of austempering carbon content of austenite increases from 1.35 wt% to 1.66wt% whereas in case of austempering of DI it increases from 1.36wt% to 1.55wt%. XDR plot (2 theta Vs. intensity) for A1, B1, A3 and B3 are plotted in figure 16.
The presence of martensite after 1h of austempering can be confirmed from (101) martensite peak at 43.44° (figure 16 (a) and (b)). As austempering time increases austenite gets saturated with carbon atom and martensitic starts temperature ($T_{MS}$) decreases below room temperature. Thus austenite is stable at room temperature. After 3h of austempering no martensite peak is observed figure 16 (c) and (d).

### 3.4 Hardness

The hardness of as cast ferritic-pearlitic microstructure of ductile iron is 28 HRC. After heat treatment of ductile iron pearlitic matrix gets converted into ferritic matrix thus reduces hardness to 6 HRC. The hardness obtained after austempering shown in figure 17.

After austempering of both irons hardness is improved. Maximum hardness (50 HRC) is obtained at B0 due to martensite. As the austempering time increases martensite disappeared from microstructure, hence hardness decreases.

In FDI, at 0h of austempering hardness is 45 HRC. After 2 h of austempering hardness reduces to 41 HRC and then remains constant. In DI, after 2h of austempering hardness reduces to 44 HRC from 50 HRC. The hardness obtained after austempering of DI is more than that of austempered FDI.
The presence of martensite at 0h of austempering is conform from SEM image, XRD plot and hardness value. As rate of austempering reaction is more in case of austempering of FDI hence more austenite and less martensite is formed. Thus more amount of austenite gets converted to martensite at B0 than A0 due to which hardness is more at B0 than that at A0.

At B5, carbon content of austenite reduced to 1.50 wt% and hardness rise to 45 HRC. This indicated that the second stage of austempering reaction starts.

4. Conclusions

The Ferrite ductile iron (FDI) was successfully produced from ductile iron by heat treatment with 70% of ferrite area fraction. Due to transformation of matrix from pearlite to ferrite hardness drops and graphite area fraction increases.

The martensite was observed after 1h of austempering time. As austenite doesn’t have sufficient time to get saturated with carbon atoms and gets converted into martensite upon air cooling after 1h of austempering. The presence of martensite after 1h of austempering was confirmed by XRD analysis and high hardness value in both DIs. As the austempering time increases austenite gets saturated with carbon atom hence weight percentage carbon content and lattice parameter of austenite increases. Due to this martensite disappeared from matrix and CSA area fraction increases.

Austempering of FDI produces finer ausferritic matrix than that obtained in austempering of DI. Ferrite cell size is less in austempered FDI than that in DI. The rate of austempering reaction in FDI is more than that of in DI. Due to this area fraction of CSA and carbon content of austenite is more in FDI than that of DI. Ausferritic matrix coarsened (feathered) with increasing in austempering time for both DIs. Hardness is improved after austempering process in both DIs due to ausferritic matrix. The highest value of hardness was 50 HRC obtained at B1. The hardness of austempered FDI is lower than in case of ADI.
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