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# **Phase Investigation of Austempered Ductile Iron**

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Abstract : In austempering, the microstructural end product of the spheroidal graphite (SG) iron matrix is essentially bainite, a structure formed below the pearlite temperature range but above the martensite range. Ductile cast iron undergoes a remarkable transformation when subjected to the austempering process. Due to isothermal transformation, it produces a microstructure that is stronger and tougher than the structures resulting from conventional heat treatment process. In the present investigation, the SG iron was austempered with three different austempering temperatures  $(250^{\circ}C, 300^{\circ}C \text{ and } 350^{\circ}C)$  with varying austempering time. The sample was taken for XRD analysis to study the morphology of the matrix. It was found that both the austenite (111) and ferrite (110) lines are identified nearly in all cases. The maximum intensity of the austenite (111) line is increasing with increasing temperature but ferrite (110) line is increasing with increasing austempering time and decreasing with austempering temoerature. Hence austempering calls for very precise control of process times and temperatures.

Keywords: Spheroidal Graphite Iron, Austempering, XRD analysis

#### 1. Introduction

Austempered ductile iron (ADI) is considered to be an important engineering material because of its attractive properties such as good ductility at high strength, good wear resistance and fatigue strength and fracture toughness [1]. Because of these combinations of properties, ADI is now used extensively in many structural applications in automotive industry, defense and earth moving machineries [1]. The optimum mechanical properties of ADI i.e., the adequate combination of strength, toughness, fatigue strength, and wear resistance could be achieved if the microstructure consists of retained carbon-enriched stable austenite (enables ductility), together with one of two bainitic morphologies, namely, carbide-free bainitic ferrite or bainitic ferrite, in which carbides are

distributed in the ferrite (affects strength)[2]. The mechanical properties of ADI depend on the microstructure, which in turn depends on the austempering variables, i.e. austempering temperature and the time of holding [4]. In conventional ductile iron the mechanical properties can be attributed to the pearlite and the ferrite present in the matrix but the superiority in the mechanical properties of the ADI are due to the acicular ferrite and carbon enriched stabilized austenite present in the matrix [2]. The proportion in which these two phases are present depends on the austempering variables.

ADI is produced by heat treating ductile cast iron. Sometimes small amounts of copper, nickel and molybdenum are added to improve hardenability. The heat treatment parameters are carefully selected for the application. Castings are first austenitised to dissolve carbon, and then quenched rapidly to avoid the formation of pearlite. Austempering brings about the nucleation and growth of acicular ferrite, accompanied by rejection of carbon, into the austenite. The resulting microstructure, known as "Ausferrite", gives ADI special properties. Ausferrite exhibits twice the strength for a given level of ductility compared to the pearlitic, ferritic or martensitic structures formed by conventional heat treatments. The mechanical properties of the austempered ductile iron are depending on the ausferrite microstructure. The austepmered matrix is responsible for significantly better tensile strength to ductility ratio than is possible with any other grade of ductile iron [3]. An unusual combination of properties is obtained in austempered ductile iron because of the ausferrite microstructure. These properties mainly depend on the heat treatment conditions and alloyed elements. Alloy additions may be made to austempered ductile iron with a view to control the matrix structure.

Advantages of ADI, in many cases, over other cast irons and cast steels have led to the need to extent limitations of the material so that those inherent advantages can be expanded throughout the manufacturing industry. Expanded usage of a material necessitates the use of technological advances in characterization of material behavior to further define that material's ability to perform within limits of the design. The mechanical properties of ductile iron and ADI are primarily determined by the metal matrix. The matrix in the conventional ductile iron is a controlled mixture of pearlite and ferrite. The properties of ADI are due to its unique matrix of acicular ferrite and carbon stabilized austenite, called Ausferrite. The austempering process is neither new

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nor novel and has been utilized since 1930's on cast and wrought steels. The austempering process was first commercially applied to ductile iron in 1972 and by 1998 world-wide production was approaching 100,000 tonnes annually [7,14,15].

# 2. Experimental Procedure

Two heats (H1 and H2) of spheroidal graphite iron were produced in a tea spout tundish ladle method. For this study charges consists of 100kg pigiron, 400kg SG returns, 500kg steel scraps,35kg coconut charcoal and 1.3kg of 75% foundry grade ferrosilicon were melted in 1000 kg capacity of coreless induction furnace as per standard procedure and foundry practices. Two kg of copper was added in second heat during melting. The melt iron was treated in ladle at 1445-1455<sup>o</sup>C with Fe-Si-Mg by plunging method to achieve Mg content in the range of 0.04 to 0.05 wt% in the melt. Proper post inoculation was also carried out during tapping and pouring of metal to ensure the high level of nodule count. The final chemistry of the treated iron was given in table 1. The treated iron was poured into a dry sand mold bonded with furan resin and catalyst to cast Y block as per ASTM 897. For each heat three molds were poured.

After 48 hours of pouring , the casting was taken and cut in to specimens (10x10x50 mm) for austempering process. The samples were austenitised to  $900^{\circ}$ C for one hour in a muffle furnace and then austempered in a salt bath (50 wt% NaNO<sub>3</sub>+50 wt% KNO<sub>3</sub>) maintaining at three different austempering temperatures  $(250^{\circ}$ C,300<sup>o</sup>C and 350<sup>o</sup>C) for half an hour, one hour and 2 hour respectively. Finally the samples were machined and polished as per standard for XRD analysis.

Heat	С	Si	Mn	S	Р	Cr	Ni	Mo	Cu	Mg
H1	3.65	2.22	0.16	0.009	0.024	0.02	0.05	0.02	0.06	0.044
H2	3.64	2.19	0.17	0.010	0.023	0.02	0.05	0.02	0.45	0.046

 Table 1. Final chemistry of treated iron (wt %).

### 3. Results and Discussions

The X-ray diffraction (XRD) analysis was performed for both the samples of H1 and H2. This technique was used to estimate the volume fractions of retained austenite and ferrite in the material after treatment. XRD was performed 30 KV

and 20 mA using a Cu- K $\alpha$  target diffractmeter. Scanning was done in angular range 2 $\theta$  from 40° to 48° and 70° to 92° at a scanning speed of 1°/min. The profile was analyzed on computer by using X" Pert High Score Software to obtain the peak position and integrated intensities of the austenite and ferrite. By comparing these intensities the volume fractions of retained austenite and ferrite were estimated. Fig.1 and Fig 2 shows the austempering process and control of time and temperature on the process.



**Fig.1:** Austempering Process [6]

Fig.2. Control of Time and Temperature on the Process [6]

The attractive properties of ADI are due to the uniqueness in its microstructure which consists of ferrite ( $\alpha$ ) and high carbon austenite ( $\gamma$ HC). This is different from the austempered steels where the microstructure consists of ferrite and carbide. The product of austempering reaction in ductile iron is often referred to as ausferrite rather than bainite[1]. The high silicon content of the ductile iron suppresses the precipitation of carbide during austempering reaction and retains substantial amount of stable high carbon austenite (yHC) [1,2]. During austempering, the bainitic ferrite forms by rejection of carbon into the residual austenite. As austempering progresses, more of bainitic transformation occurs accompanied by rejection of more carbon into the surrounding austenite resulting in the increase in the amount of austenite and the amount of carbon in the austenite. In earlier stages, the carbon content of austenite is insufficient to make it stable, and therefore, it transforms to martensite. However, at longer times austenite is enriched to the extent that it can become thermally stable to well below room temperature [7,11]. The XRD pattern of austempered ductile iron (H1) austempered at different temperatures and different times as shown in following figures (Fig 3, 4, 5, 6, and 7).

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**Fig.3:** Austempered at  $250^{\circ}$ C for one hour

Fig.4: Austempered at 250<sup>o</sup>C for one and half hour



Fig.5: Austempered at 250<sup>o</sup>C for two hours

Fig.6: Austempered at 300<sup>o</sup>C for one hour

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**Fig.7** : Austempered at  $350^{\circ}$ C for one hour

In the XRD pattern it is observed that the austenite (111) lines and ferrite (110) are identified nearly in all cases. The maximum intensity of the austenite

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(111) line is increasing with increasing temperature but ferrite (110) line is increasing with increasing austempering time and decreasing with increasing temperature. The bainitic transformation in the austempered ductile iron can be described as a two stage phase transformation reaction. The initial transformation is of primary austenite ( $\Upsilon$ ) decomposing to ferrite ( $\alpha$ ) and high carbon-enriched stable austenite ( $\Upsilon$ HC). This transformation is commonly known as the stage I reaction [2].

# **Stage I:** $\Upsilon \alpha + \Upsilon HC$

If the casting is held at the austempering temperature for too long, then a second reaction (stage II) sets in, where high-carbon austenite further decomposes into ferrite and carbide.

# **Stage II:** $\Upsilon$ HC $\alpha$ + Carbide

Stage II reaction is undesirable since it causes the embrittlement of structure and degrades the mechanical properties of ADI. The carbide formed is  $\varepsilon$  carbide which makes the steel brittle [1, 12]. Since,  $\varepsilon$  carbide is a detrimental phase constituent, hence this reaction during austempering process must be prevented. The XRD pattern of austempered ductile iron (H2) austempered at different temperatures and different times as shown in following figures (Fig 8, 9, 10, 11, and 12).



**Fig.8:** Austempered at 250<sup>o</sup>C for one hour



The best combination of mechanical properties in ADI can be obtained after the completion of the first stage reaction but before the onset of the second reaction. The time interval between the completion of the first reaction and the onset of the second reaction is termed as "process window". In order to enlarge this process window alloying elements are added [9, 10]. In the XRD pattern it is observed that the austenite (111) lines and ferrite (110) are identified nearly in all

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**Fig.10**: Austempered at 250<sup>o</sup>C for half an hour **Fig.11**. Austempered at 350<sup>o</sup>C for one hour



Fig.12: Austempered at 300<sup>o</sup>C for one hour

Alloying element (Cu) was added in order to delay the transformation of austenite in ductile iron. Depending upon the relative effectiveness of the alloying elements on the reactions in stage I and stage II of the austempering process, they are added. Cu does not alter the carbon diffusion in austenite or the stability of austenite. It has been reported that Cu suppresses carbide formation in lower bainite [5,13]. Copper widens the austenite zone of the phase diagram, increasing both the transformation rate during austenitising process and the carbon content in the matrix. Again, during the subsequent austempering process, copper may restrain carbide formation [6]. Hence, stage II reaction may be delayed due to Cu addition and result in prevention from deterioration of properties.

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### 4. Conclusions

The influence of the austempering process on the graphite morphology was discussed by XRD analysis. The effect of alloying element (Cu), austempering time and temperature on the graphite morphology of samples for the heats H1 and H2 has been investigated by XRD analysis method. The present study shows that the relation between alloying element and austempering process is an important factor for property development of ADI. The austenite is increasing with increasing austempering temperature and ferrite is increasing with increasing austempering time in both the grades (H1 and H2). The samples which are austempered at higher temperatures having upper bainitic structure and the samples which are austempered at lower temperatures are having lower bainitic structure in both the grades. From the XRD analysis it may conclude that austenite lines (111) are increasing with austempering temperature but ferrite lines (110) are increasing with austempering time and decreasing with austempering temperature. This behavior was found more prominent in H1 grade as compared to H2 grade. Samples of H2 grade will show improvement in mechanical properties as compared to samples of H1 grade.

#### References

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- [1] J Yang and S K Putatunda, Mater. Des. 25, 219 (2004)
- [2] O Eric, L Sidjanin, Z Miskovic, S Zec and M T Jovanovic, *Iron. Mater. Lett.* 58, 2707 (2004)
- [3] Chandler Harry, *Heat treaters guide: Practices and procedures for irons and steels*, IInd Sub-edition (ASM International, 1995)
- [4] O Eric, M Jovanovic, L Sidjanin, D Rajnovic and S Zec, Mater. Des. 27, 617 (2006)
- [5] J R Keough, Section IV Austempered Ductile Iron, 1998
- [6] http://www.advancedcast.com/austempering-process.htm
- [7] U Batra, S Ray and S R Prabhakar, JMEPEG. 13, 64 (2004)
- [8] Ductile Iron society, ductile Iron Data for Design Engineers. http://www.ductile.org/didata/pdf/didata2.pdf,9
- [9] J L Doong, C Chen, Fract. Engg .Mater Struct. 12, 155 (1989)
- [10] D J Moore, T N Rouns and K B. Rundamn, J. Heat Treat. 4, 7 (1985)
- [11] M Tayane, K Aztekin and A Bayram, Materials and Design. 19 (2006)
- [12] Introduction to Physical Metallurgy: Sidney H.Avner.
- [13] Physical metallurgy for Engineers: Clarke & Varney.
- [14] K K Schrems, J A Hawk, N Do-an and A P Druschitz, "Statistical analysis of the mechanical properties of thin walled DI castings."
- [15] C Labre and M Gagnj, AFS Trans. 108, 31 (2000)