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Composition and Austempering Heat Treatment on Ductile Iron

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Abstract: Austempered ductile irons (ADI) is the developed material of the DI family. By adapting the austempering treatment initially introduced DI; it has been shown that the resulting metallurgical structures provide properties that favorably compare to those of steel while taking advantage of a near-net-shape manufacturing process. Here the desired composition and production of Ductile were clearly studied which is suitable for heat treatment also. The Effect of hardenability agents which is more wanted for the austempering treatment and their optimized values are given along with disadvantages. The austempering heat treatment parameters and the effect of mechanical property were studied along with microphotographs.

Keywords: Solid state transformation, intercellular carbides, austempering time.

I. INTRODUCTION

Austempered Ductile Iron (ADI) results from a specialty heat treatment of ductile cast iron. Strength improvements up to 100% (or more) in combination with excellent toughness can be realized by using this process. Successful production of ADI requires a cooperative effort between the foundry and heat treater. High quality ductile iron is the necessary raw material. As a family of materials, austempered ductile iron capably addresses the issues of weight, strength, stiffness, fatigue toughness, design flexibility, noise, low cost and recyclability. From the first differential gear sets installed by General Motors in 1978, to light- weight truck-trailer wheel hubs, to high performance automobile suspensions, crank and cam shafts austempered ductile iron has found itself in many unique applications.

ADI has been developed to the point where foundry men, heat treaters and design engineers have sufficient technical information to take full advantage of the engineering properties of the material. Nevertheless, as for other engineering materials, the production parameters have a significant influence on the performance of ADI castings. Of particular importance is the as-cast quality of the castings submitted to austempering as the heat treated castings remain sensitive to casting defects. The purpose of this paper is to review the metallurgical aspects of ADI production in the foundry as well as in the heat treating shop.

II. Ductile Iron Casting for ADI Production

Production

Figure.1 presents a typical flow sheet for the production of DI castings. For ADI castings as for all other DI grades, the charge material must be carefully selected to prevent the contamination of castings and defects by certain alloying elements found in steel and maintain a high level of consistency of the microstructure.

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Table 1 lists the recommended limits for some of the most common detrimental residual elements found in DI castings (e.g. S, P, Mn, Cr and Cu).

Figure.1.Steps for the production of DI

Although every production step outlined in Figure.1 is important to ensure the production of defect free castings. Special attention must be paid to the spherodization and inoculation practices, which control the crystallization of spherulitic graphite particles. In order to obtain high quality DI components, the maximum possible level of these must be achieved.

Composition

When compared to gray iron, the sensitivity of a DI component to its chemical composition is very high. In gray iron the graphite flakes are the weakest structural features whereas in DI castings it is defects often caused by elements present in the charge materials (e.g. eutectic and intercellular carbides, flake graphite,..) that limit the performance. Therefore, the careful selection of the charge materials is critical.

Element	Suggested Target
Carbon – C	3.6%
Silicon – Si	2.5%
Phosphorus – P	0.04% maximum
Magnesium– Mg	(%S x 0.76)+0.025%
Sulfur – S	0.02% maximum
Manganese – Mn	0.35% maximum
Max section>13 mm	0.60% maximum
Max section<13 mm	0.0070 maximum
Copper – Cu	0.80% maximum
Nickel – Ni	2.00% maximum
Molybdenum - Mo	0.10% maximum
Tin - Sn	0.02% maximum
Antimony – Sb	0.002% maximum
Oxygen – O	50 ppm maximum
Chromium – Cr	0.10% maximum
Titanium – Ti	0.040% maximum
Vanadium – V	0.10% maximum

Table 1: Suggested Targets for the Production of ADI

Table.1 lists the recommended ranges of chemical composition for the DI grades. Although not mandatory, a peartic matrix is usually recommended for ADI production in order to facilitate the dissolution of carbon in austenite during the heat treatment. Therefore, the carbon and silicon contents should be adjusted as a function of the casting section size, as shown in table.2. For castings above 12mm wall thickness, the addition of pealite promoters which are not carbide formers, such as copper or tin, is usually required.

Wall Thickness	Carbon (wt%)	Silicon (wt%)	Carbon Equivalent
3	3.90	2.90	4.87
6	3.85	2.65	4.73
12	3.70	2.45	4.52
25	3.60	2.35	4.38
50	3.45	2.20	4.18
100	3.40	2.15	4.12

Table:2 Contents for pearl	itic Ductile iron castings
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Ductile iron castings destined for ADI production need to be alloyed with hardenability enhancing elements, the most common being Cu, Ni, Mo, Mn. The two latter are however potent carbide formers and as such, their concentrations must be limited as will be shown in section IV. Nonetheless, Mo is usually preferred to Mn because of its stronger hardenability enhancement effect for an equivalent intercellular carbide forming tendency.

Microstructures and Properties

As shown in Figure 2, the distinctive characteristics of DI is the presence of spheroidal graphite in the pearlite matrix.



Figure.2. Photomicrograph of DI.

The ductility and the property of the casting depend on the shape and number of casting per unit area. The higher the number of spheroids (or nodule count), the greater the quality of castings; a high nodule count, which is main, controlled by the inoculation process, limits the chemical segregation during solidification and ensures the structural homogeneity of the component. A minimum of 80% nodularity is usually easily achieved. Also the graphite nodules must be as spherical as possible, since irregular graphite particles act as stress raisers.

It is generally accepted that one should produce castings with the consistent graphite characteristics and then control the mechanical properties by the microstructure of the matrix. In the as-cast condition .the properties of a defect-free unalloyed castings will be governed by the ferrite/pearlite ratio. However, via alloying and/or heat treatment, the matrix structure can be altered to obtain the desired properties. Figure. 3 illustrates the range of properties achievable for given microstructures.



Figure. 3. Range of properties of DI family.

III. Manufacture and Characteristics of ADI Castings

Heat Treatment and Microstructure

A schematic of the austempering heat treatment process is shown in the Figure.4. This treatment consists of



Figure.4. Austempering Process

- 1. Complete austenization of the castings;
- 2. Quenching to austempering which is lower than the pearlitic transformation temperature but higher than that for martensitie transformation in a salt bath containing sodium nitrate & potassium nitrate of equal Wt%.
- 3. Holding for a given time at the austempering temperature followed by cooling to a room temperature.

Because of the high concentrations of carbon and silicon in DI, the solid-state transformation of the austenite during austempering differs from that observed in steels. Figure.5. presents schematically the structural changes occurring during austempering. Individual plates of ferrite, separated from each other by thin layers of carbon saturated austenite, nucleate at austenite grain boundaries and grow. As the reaction proceeds, the carbon diffusion ahead of the ferrite plates become more difficult and the growth of the plate ceases, resulting in a duplex ferritic-austenitic matrix, called ausferrite. The high carbon content of DI favours the formation of such a structure by slowing down the growth of the ferritic plates and stabilizing the austenite. Because austenite is metastable at low temperature, it will eventually transform to ferrite and very fine globular cementite. This transformation is however delayed by the high silicon content of the alloy which retards carbide formation. Therefore, the austempering reaction can be considered as a two stage solid-state transformation of austenite.

 $\begin{array}{l} \gamma 0 \rightarrow \alpha + \gamma H.C \\ \gamma H.C \rightarrow \alpha + Fe3C \\ When \\ \gamma 0 \ \ is the primary austenite and \\ \gamma H.C \quad the carbon enriched austenite. \end{array}$

Note that the optimum mechanical properties are attained before the onset of the second reaction. The effect of the heat treatment parameters on the kinetics of the austempering reaction will be reviewed in section VI.



Figure.5. Solid-State transformation of austenite during austempering

Properties

The particular microstructure of ADI offers a unique combination of tensile and fatigue strengths, ductility, toughness, wear resistance and machinability in conjunction with design flexibility and low cost. Not only does ADI outclass the properties of other DI grades as shown in Figure. 3, it also compares advantageously with forged steels. Moreover, the strength-to-weight ratio of the material is among the lowest of all engineering materials, including aluminum.

Table.3. lists the five ADI grades standardized by ASTM A897 / 897M. The strength/ductility combination can be varied by changing the ausferrite microstructure through careful selection of the heat treatment parameters.

Grade	Tensile Strength (MPa/Ksi)	Yield Strength (MPa/Ksi)	Elo (%)	Impact Energy (J/ft-lb)	Hardness (BHN)
1	850/125	550 / 80	10	100 / 75	269-21
2	1050/150	700 / 100	7	80 / 60	302-363
3	1200/175	850 / 125	4	60 / 45	341-444
4	1400/00	1100 / 155	1	35 / 25	366- 77
5	1600/30	1300 / 185	NA	N/A	444 - 55

Table.	3. ADI	Grades.
140101		Gradoo.

A significant advantage of ADI is its outstanding wear resistance due to the strain induced transformation of the un-reacted high carbon austenite that occurs when the surface of an ADI casting is subjected to deformation. As shown in Figure.6.



Figure.6. Microhardness of ADI Sample.

This transformation results in significant increase of surface hardness with the bulk of the part retaining its ductility. Such a surface strain-hardening effect permits the continuous maintenance of a high surface hardness by the uninterrupted replacement of the abraded layer. Finally inspite of its high strength and wear resistance, ADI offers excellent machinability when compared to competitive materials with equal properties.

IV. Effect of Production Parameters on ADI Characteristics

Role of Alloying Elements

The primary role of alloying elements in ADI is to provide sufficient hardenability to the matrix to avoid the pearlite nose of the CCT curve, as illustrated in Figure.4. However, this must be achieved with a minimum effect on the first stage of the ausferrite transformation; an alloying element might delay the austenite stabilization, which may result in the transformation of the austenite to martensite after cooling to room temperature. For various reasons, either economic or quality- related a combination of alloying elements, usually Mo, Ni, Cu and Mn is used.

Manganese

It has been reported that manganese decreases the UTS and elongation. Effect of Manganese on the impact values are specified in the table.4.

Mn (%)	Impact Value (J/cm ²) (Charpy)
0.07	78.5
0.47	55.9
0.74	36.3

Table.4. Effect of Mn on the impact Value

The manganese content in ADI content should not exceed 0.5%. Beyond this limit, the as-cast qualities of the part as well as the kinetics of the solid state reactions occurring during austempering are affected.

As regards the initial quality of castings, manganese has a twofold effect. In small section castings, it has been shown to favor the formation of eutectic carbides, whose presence requires either a longer austenitizing time or an annealing of casting prior to austempering. In castings solidifying at intermediate or slow rate, manganese segregates at cell boundaries causing the precipitation of intercellular complex Fe-Mn carbides, as illustrated an Figure.7.



Figure. 7. Manganese alloyed intercellular Carbides.

These manganese- stabilized carbides usually remain as a highly brittle phase in the structure after austempering treatment.



Fig.8.(a) Intercellular Features – Martensite.



Figure.8.(b) Intercellular Feature–Untransformed austenite.

Segregation of manganese at the cell boundaries also results in a heterogeneous hardenability of the matrix by increasing the stability of the intercellular austenite. These segregated regions may either transform to martensite after cooling from the austempering temperature. Figure.8 (a), or remain as large unreacted austenite areas, Figure.8(b).



Figure.9. Effect of Manganese on the austempering Time

Figures 9 and 10 shows, respectively, the effect of manganese content on the quality of such inter-cellular phases in the austempered component and on the austempering time needed to avoid martensite formation. These intercellular phases severely affect the mechanical properties of ADI castings, as illustrated for fracture toughness in Figure.10.



Figure .10. Effect of Mn & temperature on fracture toughness.

Molybdenum

Molybdenum is the most potent hardenability agent in ADI. However, like manganese, molybdenum segregates at cell boundaries during solidification to form carbides. These Mo-rich carbides serve as nucleation sites as well as propagation paths for cracks. Both phenomena are illustrated in Figure.10 for an ADI casting containing 3.6%C, 2.5Si, 1%Cu, 0.2% Mn and 0.25% Mo which reputed after only a 2% tensile elongation.



Fig. 10. Crack initiation and propagation on Mo-Fe carbides

The white phase revealed by back scattered electron microscopy was analyzed as complex iron-molybdenum carbide. The use of molybdenum should thus be limited to the minimum concentration needed to obtain the required hardenability for a given section size. For practical purposes, molybdenum should be used in the combination with nickel and/or copper and its concentration should not, in any situation, exceed 0.2%.

Copper and Nickel: Copper addition alone does not provide sufficient hardenability to successfully austemper a casting of 2.5 cm diameter, although this could achieved by the combining nickel and copper. A material of such composition would however contain about 15% pearlite at the centre of a 5.1 cm diameter austempered casting but be fully pearlitic at 0.5 cm under the surface of a 7.6 cm diameter casting. Increasing the nickel and/or copper contents improves the hardenability, as shown in Figure.11, but not sufficiently to avoid pearlite formation at the centre of 5.1 cm diameter castings.

The addition of small quantities of Mn and/or is necessary to fully austemper such castings (see Figure. 11). Major advantages of using nickel and copper in order to minimize the concentration of Mn and/or Mo are:

- i. To avoid the segregation of the latter elements to the cell boundaries,
- ii. And, as shown in the Figure. 12, to shorten the time required completing the austempering reaction.



Figure.11. Effect of cu and Ni on pearlite fraction austempered 1h at 3600C.



Figure .12. Effect of austempering time on the presence of martensite in austempering 1h at 3600C

Austempering Treatment Parameters

Austenitizing: The austenitization of ductile iron castings of suitable chemical combination is the first step of the ADI treatment. The austenitizing temperature controls the carbon content of the austenite which in turn affects the structure and properties of the austempered castings. For example: austenitizing at 8500C or 9000C results in austenite carbon content at equilibrium of 1.1 or 1.3%, respectively. A higher carbon content of the austenite increases its hardenability and stability, and affects the austempering reaction. As shown in the Figure 13. austenitizing at 9000C delay the first stage of the austempering reaction and may result in the formation of martensite if the austempering time is too short. However, austenitizing at higher temperature favors the redistribution of segregated elements and the decomposition of intercellular carbides when present.



Figure.13.Effect of austenitizing temp. on the amount of untransformed austenite in ADI castings.

The austenitizing time should be the minimum required to ensure that the casting has been completely transformed to carbonsaturated austenite. One hour is usually appropriate at 9000C for a 2.5cm diameter casting. Nevertheless, at lower temperature up to 3 h are recommended to ensure the reduction of the micro segregation of alloying elements. As for steels, the austenitizing time and temperature have to be adjusted as a function of the section size.

Austempering: As previously indicated, the hardenability of the ductile iron casting to be treated and the severity of the cooling from austenitizing to the austempering temperatures must be sufficient with regards to the casting section size to avoid the formation of pearlite. When this is achieved, the structure and properties of the casting will be determined by austempering at a given temperature for an optimized time.

Table.3. Presents the property ranges of the five ASTM standard ADI grades. The tensile strength and elongation levels, which vary from 850 MPa and 10% to 1400 MPa and less than 1%, are determined by the austempering temperature. The typical temperature ranges utilized are $460 - 750^{\circ}$ F (or 238 - 399°C). The lower grades (1 and 2) require temperature choices at the upper end of the range while the higher grades are produced at lower quench temperatures.

Time at temperature is dependent on the choice of temperature as well as the alloy content. For example, Grade 1 ADI will transform faster than Grade 5 as the quench temperature is approximately $200^{\circ}F$ (93°C) higher.

The components are held for a sufficient time at temperature for ausferrite to form. Ausferrite consists of ferrite in a high carbon, stabilized austenite. If held for long time periods, the high carbon austenite will eventually undergo a transformation to bainite, the two phase ferrite and carbide (α . + Fe3C). In order for this transformation to occur, longer periods of time are typically needed – much longer than would be economically feasible for the production of ADI.

Once the ausferrite has been produced, the components are cooled to room temperature. The cooling rate will not affect the final microstructure as the carbon content of the austenite is high enough to lower the martensite start temperature to a temperature significantly below room temperature.

Figures 14(a) and (b) show the ausferrite microstructure for Grades 1 and 5 ADI, respectively.

Such effects of the austempering temperature are related to microstructural changes. as shown in Figure .15(a), a high austempering temperature (3600C) results in a coarse micro-structure with a fairly large amount of austenite.



Figure 14.a: Photomicrograph of Grade 1 ADI. Specimen was etched with 5% Nital.



Figure 14.b: Photomicrograph of Grade 5 ADI. Specimen was etched with 5% Nital.

Reducing the austempering temperature of 3100C favors the formation of finer microstructure with smaller isolated austenite islets in a large undercooling of the austenite and a slow diffusion rate of carbon, the nucleation of ferritic plate rather than their growth is favored, resulting in a finer structure.

Once the austempering temperature has been selected to obtain the desired properties, the treatment time has to be optimized. Martensite forms in the austempered structure after short tempering times because of either the higher hardenability of Mn-segregated areas and/or because of the lower carbon content of some austenitic regions.

This is shown in Figure. 16. which presents the martensite content in the heat-treated structure of various alloys after austempering 0.5 h at either 310 or 3600C. it is seen that martensite, although in very small quantities, is almost unavoidable in all alloys when treated for only 0.5h. However, for both austempering temperatures the presence of manganese results in up to 40% martensite in the structure, clearly indicating that the minimum austempering time must be increased because of the presence of manganese. Therefore, the austempering has to be sufficiently long to eliminate the untransformed austenite and consequently the martensite that might form.





Figure.15. Effect of austempering temp on the mocrostructure of ADI after 1h austempering. (a) Temperature: 3600C, (b)Austempering Temperature: 3100C

As shown in Figure.12, in absence of manganese, 1h austempering is sufficient; this minimum time increases to 2 h for 0.2% Mn alloyed castings. Although a long austempering time ensures the avoidance of martensite due to the presence of manganese, large unreacted austenitic areas may remain and embrittle the castings.



Figure.16. Effect of chemical composition & austempering Temp.

Finally, an extreme austempering time would cause the decomposition of the stabilized austenite to ferrite and carbides, as seen in Figure.13, making the structure brittle.



Figure.17. Effect of austempering Time on the Mechanical properties.

Usually, for a casting containing less than 0.2% Mn, a 1-2 h austempering time is appropriate, as confirmed by the evolution of the tensile properties as a function of the austempering time which is presented in Figure 17.

Concluding Remarks

Austempered ductile iron exhibits a unique microstructural arrangement of ferrite and austenite (ausferrite) conferring to the structure remarkable mechanical properties and wear resistance. However, optimum properties can be obtained only when defect-free DI castings are treated and when an adequate control of chemical composition the heat treatment is achieved with following conclusions.

- The manganese content should not exceed 0.5%.
- Fracture toughness increases with Mn.
- One hour austenizing is usually appropriate at 9000C for a 2.5cm diameter casting.
- 1-2 h austempering time is appropriate.
- Increase of austempering Time has No effect on Mechanical properties.

The combination of strength, ductility, fatigue resistance, makes ADI may substitute for steel (cast, forged and/or heat treated) or aluminium in applications where strength/weight ratio is important. Major applications include gears, crank shafts, sprocket, pinion, and many other parts used in automotive, earthmoving, and excavating and agriculture equipment.

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