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Characteristics of the Transformations Occurring within the Intercritical Interval of Ductile Iron

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Abstract. Austempered Ductile Iron (ADI) and fully ferritic ductile iron are frequently used to produce safety parts. A new kind of ductile iron (DI), usually referred to as "dual phase ADI" is currently under development. The matrix of this new material is composed of ausferrite (regular ADI microstructure) and free (or allotriomorphic) ferrite. This combined microstructure is obtained by subjecting DI to heat treatments comprising incomplete austenitization stages at temperatures within the intercritical interval (three phase α-γ-Gr field of the Fe-C-Si stable diagram) followed by an austempering step in a salt bath in order to transform the austenite into ausferrite.
This work describes some alternatives to control morphologies and amounts of phases present in the final microstructure. A revision of the results obtained on this matter by the authors as well as by other researchers is also provided. A ductile iron melt was produced in a metal casting laboratory. Samples were heat treated following different thermal cycles to obtain a wide range of duplex microstructures, containing different amounts and morphologies of microconstituents.
The results account for the fact that the transformations occurring within the α-γ-Gr field are strongly affected by the chemical composition, previous matrix, and holding times. The role played by the microsegregation and availability of sites for heterogeneous nucleation is also worth noticing. Starting from ferritic matrices and holding the samples at constant temperature within the intercritical interval, austenite nucleation and growth take place mainly in the last to freeze zones. On the other hand, when starting from fully recrystallised austenitic matrices, allotriomorphic ferrite can precipitate during the isothermal holding within the intercritical interval at the austenite grains boundaries forming a continuous net. The kinetics of the $\gamma \rightarrow \alpha$ transformation is notoriously slower than the $\alpha \rightarrow \gamma$ one. The influence that part size exerts on the final microstructure is also analyzed.

Introduction

Intercritical Interval. The thermodynamic equilibrium of phases in cast irons with free carbon are commonly represented by means of pseudo binary diagrams which are cuts from the F-C-Si ternary diagram, as sketched in Fig. 1 [1]. At temperatures close to the eutectoid, the diagram displays a three phase field where ferrite (α), austenite (γ) and graphite (Gr) coexist. This region is delimited by the lower and upper critical temperatures and is called "intercritical interval" by the authors. Such temperatures define the starting point in which ferrite transforms into austenite and austenite into ferrite in heating and cooling processes, respectively. The position and amplitude of the interval is modified by the chemical composition of the alloy. For instance, the presence of Si leads to an increment of both critical temperatures. This is explained by the fact that this element diminishes the solubility of C in austenite, thereby promoting ferrite formation. In addition, other elements, such as Mn, can lead to a decrease of the critical temperatures. The literature reports equations designed to calculate the upper critical temperature, taking into account the influence of some common alloying elements [2,3]. There are also several studies regarding the influence of continuous heating or cooling on critical temperatures (2,4). To the best of the authors' knowledge less attention was putted on the study of the metallographic characteristics of the transformations taking place at constant temperatures within the three phase field, as well as on the final microstructures obtained by an ending fast cooling step (quenching in water or salt).

It is worth mentioning that the metallic matrix of ductile iron does not feature a homogeneous concentration of the chemical elements. Significant variations in concentration exist due to the segregation produced during the solidification process. One of the main parameters determining the characteristics of the segregation profiles and the extension, distribution and morphology of the last to freeze (LTF) zones is the cooling rate imposed during solidification. It is well known that when the cast part thickness increases, a lower amount of nodules of higher size is obtained, while LTF zones enlarge exhibiting also greater concentrations of alloying elements of direct segregation tendency [5,6]. Given the fact that the position and amplitude of the intercritical interval is dependent on the chemical composition present in each position of a cast part, critical temperatures are influenced by the microsegregation occurring during solidification, and hence by the solidification rate (part size) [7].

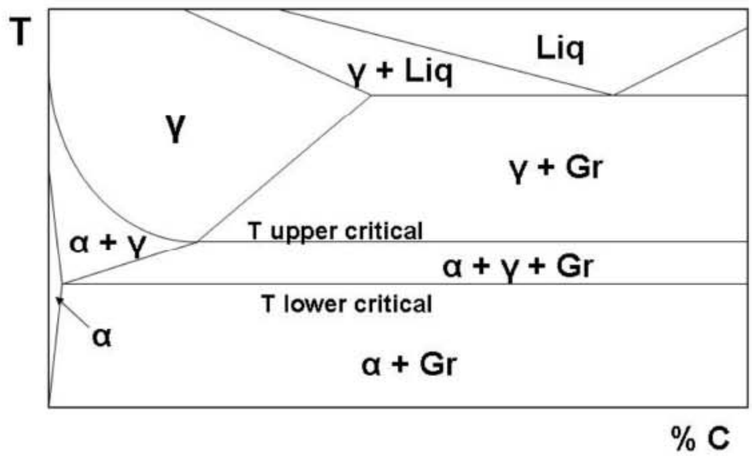

Fig.1: Schematic representation of Fe-C phase diagram (at 2.5% Si) [1].

Dual Phase Austempered Ductile Iron (dual phase ADI). The concepts tied to the intercritical interval have gained interest in the field of cast irons with duplex matrix [3,8], and nowadays especially on the development of a new variant of DI called "dual phase ADI" [9]. The matrix of this DI is composed of ausferrite and free (or allotriomorphic) ferrite. This combined microstructure can be obtained by subjecting DI to heat treatments (HT) comprising an incomplete austenitization step (by isothermal holding at temperatures within the intercritical interval) followed by an austempering stage in a salt bath, so as to transform the austenite into ausferrite [9-11]. Several studies have explored the influence of the relative amount of phases present in the matrix, and the results have demonstrated that it is possible to obtain a wide spectrum of mechanical properties, yielding an interesting strength / elongation ratio for certain microstructures, if compared to those of completely ferritic or ausferritic matrices [9-13]. The goal of performing an intercritical HT is that of obtaining a microstructure composed of well controlled amounts of ferrite and austenite with different morphologies, depending on the HT cycles and parameters selected. Precise understanding of the transformations taking place within the intercritical interval should then be developed in order to produce DI with duplex microstructures. This work analyses some aspects of the solid state transformations occurring in the intercritical interval on the basis of metallographic evidences.

Experimental Methods

A ductile iron melt was prepared in a medium frequency induction furnace. Steel scrap and foundry returns were used as raw materials. Nodulization was conducted applying the sandwich method, employing 1.5% of Fe-Si-Mg (6% Mg), while inoculation was performed using 0.6% Fe-Si (75% Si). The melt was poured in 1 and 3 inch thick "Y" blocks shaped sand moulds. The chemical composition was determined by using a Baird DV6 spectrometer. Round samples of 12 mm diameter and 30 mm length were cut from the "Y" blocks and used to prepare test specimens. Microstructure was analyzed using an Olympus G7 optical microscope and the amount of phases was quantified by means of the Image Pro Plus software. The methodology employed to establish the intercritical interval (upper and lower critical temperatures) had been previously developed by the authors [9,10]. The study of solid state transformations taking place during heating and cooling inside the intercritical interval was undertaken by using two types of HT, as sketched in Fig. 2.

Heat Treatment T1. This treatment allows studying the characteristics of the reaction $\alpha \rightarrow \gamma$ during heating, starting from a fully ferritic matrix. Cycle T1 consists essentially of an incomplete austenitising stage by holding the sample at constant temperature within the intercritical interval during one hour, followed by water quenching (see Figure 2). The final microstructures after HT are composed of different percentages of ferrite and martensite (quenched austenite) depending on the selected intercritical temperature. The effect of part size on the intercritical interval was analyzed using this HT. Two sets of samples with fully ferritic matrices obtained from 1 and 3 inch "Y" blocks were heated at 760 and 800ºC during one hour and then water quenched.

Heat Treatment T2. The aim of this treatment was to evaluate the $\gamma \rightarrow \alpha$ transformation during isothermal holding in the intercritical interval. The methodology consisted in a previous complete austenitising stage at 910ºC during one hour, followed by a rapid cooling into a salt bath at 800ºC (inside the intercritical interval), in order to nucleate and growth ferrite on the fully austenitic matrix. With a view to assessing the kinetics of the reaction, several samples were extracted at one hour intervals, followed by water quenching to transform the remaining austenite into martensite (see Figure 2). Once again, the phases present at room temperature were ferrite and martensite.

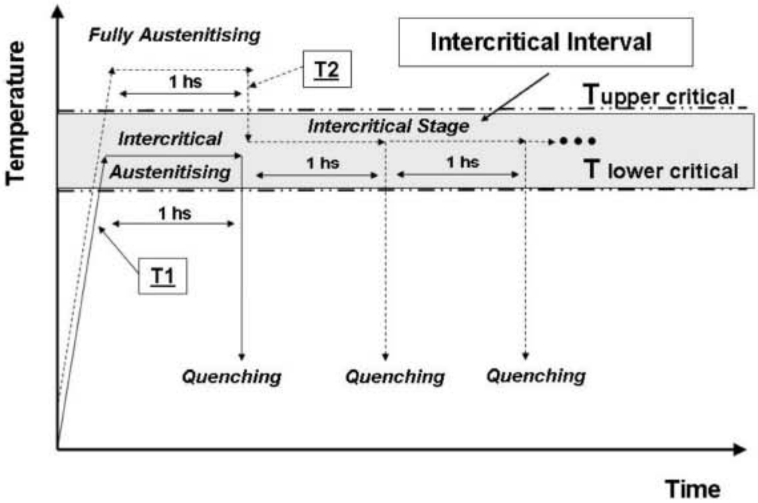

Fig. 2: Schematic of the T1 and T2 thermal cycles.

Results and Discussion

Table 1: Chemical composition of the melt.

%C	%Si	%Mn	%S	%P	%Mg	%Cu	%Ni	%CE.
3.3	2.5	0.37	0.03	0.04	0.070	0.65	0.87	4.1

Table 1 lists the chemical composition which yielded a slightly hypoeutectic melt. The microstructure in the as-cast condition resulted mainly pearlitic, with some few ferrite located around graphite nodules (bull's eye structure). Nodule counts were 200 and 50 nod/mm^2 for 1 and 3 inch thick "Y" blocks, respectively; and nodularity exceeded 90% in all cases (ASTM A-247).

Heat treatment T1: *study of the α → γ reaction.* In all cases the starting matrix was ferritic (obtained by previous annealing) with less than 5% of globullized pearlite, located in LTF zones. The lower and upper critical temperatures for the melt were established as 760ºC and 860ºC, respectively. Microphotographs in Fig. 3 depict the morphology and the amount of phases present in the microstructure after the heat treatment performed by holding the samples for one hour at some selected temperatures within the intercritical interval. Is important to point out that in previous studies the authors have proved that for this type of thermal cycle one hour is enough to reach the thermodynamic equilibrium in the α-γ-Gr region [9]. Figs. 3-A and 3-B illustrate the fact that austenite nucleation starts mainly at LTF (marked by circles in the picture). This is attributed to the higher concentration of Mn present in these zones [7,9]. Austenite nucleation and growth continues along ferrite grain boundaries (Fig. 3-C), because carbon diffusion is a process easily occurring at grain boundaries. When increasing austenitising temperature, the reaction also takes place inside the ferrite grains, which are the last zones transforming into austenite (Fig. 3-D).

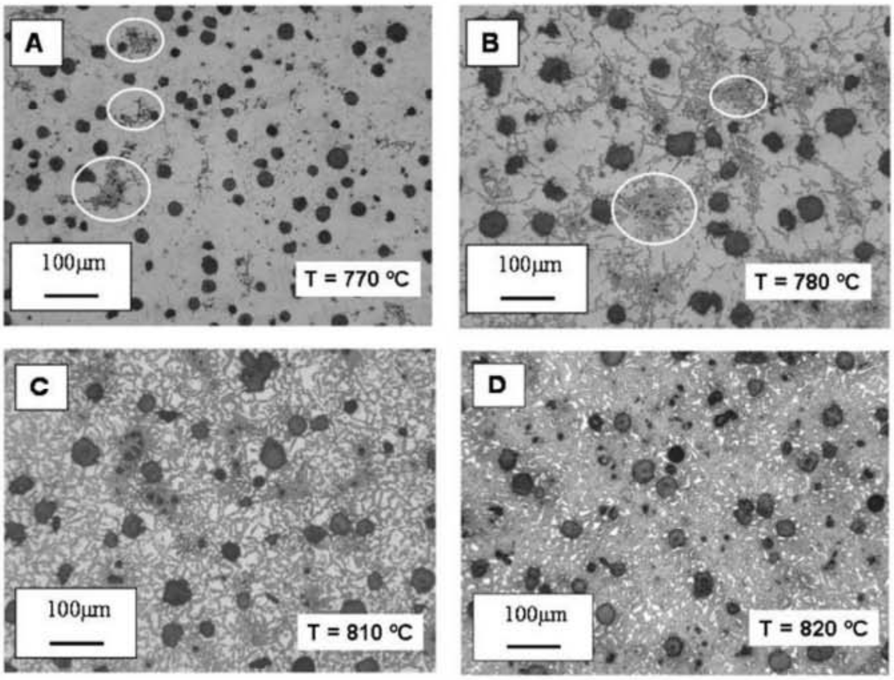

Fig. 3: Morphology and amount of phases after heat treatment T1 for different temperatures : A: 80% α - 20% γ; B: 60% α - 40% γ; C: 20% α - 80% γ; D: 5% α - 95% γ. White circles in A and B show martensite (quenched austenite) at LTF.

Heat treatment T2: *study of the γ → α reaction.* Fig. 4 illustrates the advance of the γ → α reaction as a function of holding time at a constant temperature of 800ºC (inside the intercritical interval). The nucleation of the ferrite phase starts mainly at austenite grain boundaries and at austenite-graphite interphases (Figure 4-A). This is attributed to the high energy of these zones which turns them into suitable sites for heterogeneous nucleation. On the other hand, to allow the reaction γ → α to occur, the carbon content of the austenite has to migrate to the graphite nodules, which act as carbon sinks. Then, austenite grain boundaries act as preferred sites for ferrite formation due to the high diffusivity in these zones. The interphases austenite-graphite nodules operate on the same basis, given the short length carbon needs for diffusion. Consequently, ferrite growth develops mainly at the austenite grain boundaries leading to the formation of a continuous ferrite net (Figure 4-B). Particularly worth of mention is that the morphology of the free ferrite produced by applying the thermal cycle T2 (continuous net) is fairly different when compared to that observed in as-cast microstructures generally presenting bull´s eye aspect. This notoriously morphologic change obeys to the fact that the austenite formed during heating (recrystallised austenite) nucleates in a high number of sites of the microstructure, conducting to a significantly lower grain size. The austenite grain size produces substantial differences in the nucleation process of new phases during solid state transformations [5,6]. In summary, the nucleation and growth of ferrite from recrystallised austenite starts mainly at grain boundaries (due to their large amount) and at graphite - austenite interphases. On the other hand, ferrite nucleation starting from non recrystallised austenite (very large solidification grains) occurs exclusively around graphite nodules [3,5,6].

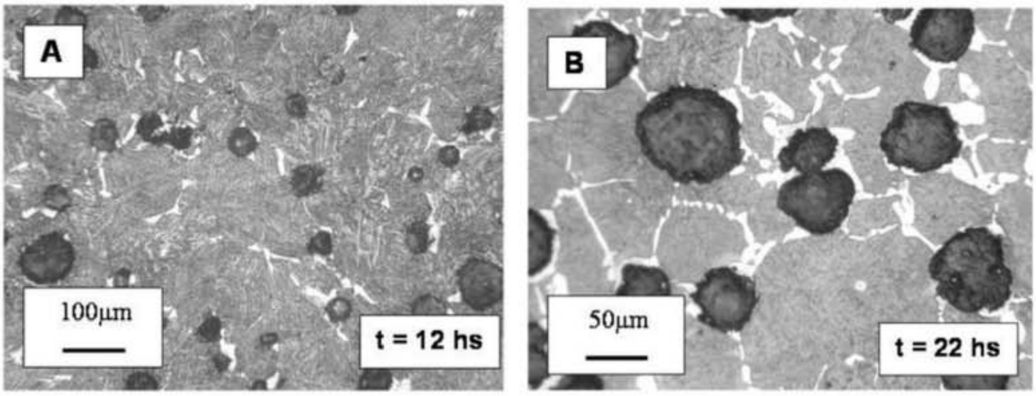

Fig. 4: Advance of the $\gamma \rightarrow \alpha$ reaction as a function of holding time at a constant temperature of 800°C within the intercritical interval.

Influence of Part Size on the Intercritical Interval Incomplete austenitization temperatures, i.e., 760 and 800°C, were used. The reason for such selection is that 760°C is the lowest intercritical temperature determined by using samples taken from 1 inch "Y" blocks, while 800°C is an intermediate temperature within the intercritical interval. Fig. 5 shows the microstructures obtained after the heat treatments (following heat treatment T1).

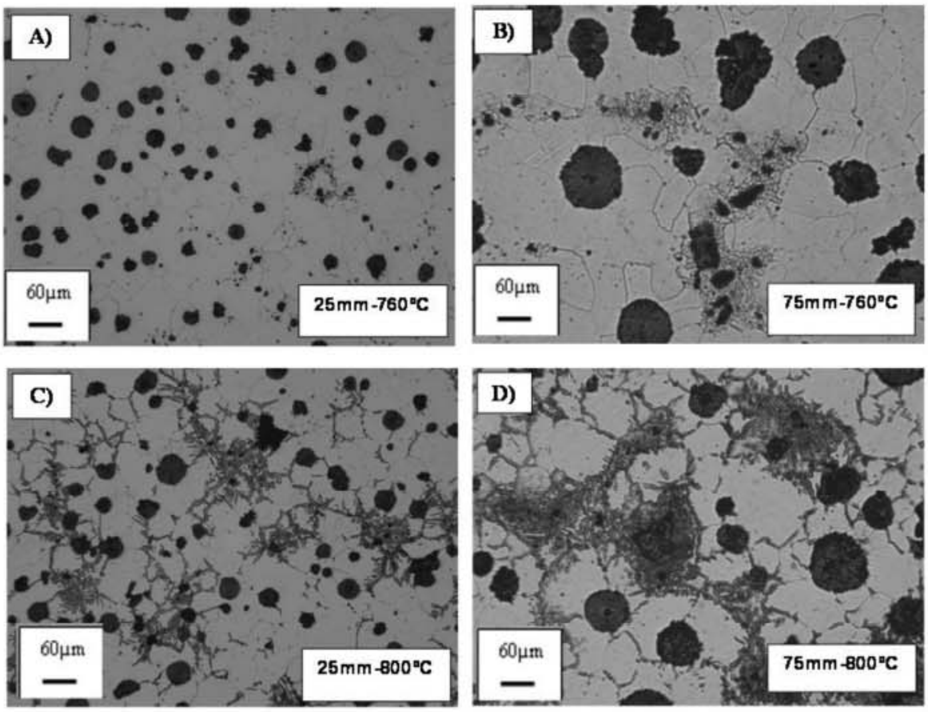

Fig. 5: Effect of section size in the intercritical interval (holding time = 1 hour)

The sample taken from the 1 inch "Y" block heated at 760°C presents no signs of transformation. This means that the HT was conducted below the lower critical temperature. Nevertheless, regarding the sample taken from the 3 inch "Y" blocks and also heated at 760ºC, some areas of martensite can be clearly observed at LTF zones (Fig. 5-B). This can be attributed to the lowering of the intercritical temperatures at LTF due to Mn microsegregation. Figs. 5-C and 5-D, in turn, depict the microstructures corresponding to the set of samples heated at 800ºC. When comparing both figures, a noticeably greater advance of transformation can be observed in the sample taken from the 3 inch "Y" blocks (Fig. 5-D). The differences answer to the effect exerted by the solidification rate on microsegregation and hence on the transformations. Then, the nucleation and growth of austenite within the intercritical interval begins at LTF because these regions feature a larger amount of segregated elements (e.g. Mn). The increase of Mn diminishes the lower critical temperature and the $\gamma \rightarrow \alpha$ reaction takes place at inferior temperature. Then, for constant chemical composition, the lower and upper critical temperatures will differ depending on the matrix site (LTF or bulk) as well as on the local cooling rate of the part concerned, demonstrating the relevance of addressing these issues whenever dual phase ADI parts of different thicknesses are manufactured.

Conclusions

1. Solid state transformations taking place inside the intercritical interval were studied. Several parameters were analyzed such as preferential sites for nucleation and growth of different phases.
2. The $\alpha \rightarrow \gamma$ transformation (cycle T1) is notoriously faster than the $\gamma \rightarrow \alpha$ reaction (cycle T2).
3. For the case of the $\alpha \rightarrow \gamma$ transformation, the nucleation and growth of austenite begins at LTF zones, continues at ferrite grain boundaries and end at the grain centers.
4. In the case of the $\gamma \rightarrow \alpha$ reaction (cycle T2) the formation of a thin net of ferrite at austenite grain boundaries can be observed. This morphologic difference with respect to the free ferrite obtained by cycle T1 and also as cast conditions has been worth pointed out.
5. The differences on the cooling rate produced by the part size modify the homogeneity of the chemical composition and therefore critical temperatures. Nucleation and growth of austenite within the intercritical interval (cycle T1) begin at LTF zones due to the higher content of gamagenous elements (Mn). Therefore, as the part size increases, the lower critical temperature diminishes at LTF zones, so favoring the transformation of ferrite into austenite at minor temperature.

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