INTERCRITICAL HEAT TREATMENTS IN DUCTILE IRON AND STEEL

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ABSTRACT

Materials such as dual phase (DP) steels, transformation induced plasticity (TRIP) steels and dual phase ductile irons are produced by intercritical heat treatments. These materials can provide significant weight savings in the automotive industry. The goal of this dissertation is to study intercritical heat treatments in ductile iron and steel to optimize the production parameters. Three different aspects were addressed.

First, common steels were intercritically austenitized and austempered (intercritically austempered) under a variety conditions. The results showed that common grade steels that were intercritically austempered exhibited tensile properties in the same range as DP and TRIP steels.

The second study consisted of determining the effect of heat treatment conditions on the tensile properties of intercritically austenitized, quenched and tempered ductile iron (IAQ&TDI). The results showed that (1) ultimate tensile strength (UTS) and yield strength (YS) were determined by the volume fraction of martensite, (2) tempering improved the elongation 1.7-2.5 times with only a slight decrease in strength, (3) the carbon in austenite formed during the intercritical heat treatment of ductile iron with a ferritic-pearlitic matrix came from the carbon available in the matrix and that carbon diffusion from the graphite nodules was restricted, and (4) limited segregation of substitutional elements occurred during intercritical austenitizing.
Finally, intercritically austempered ductile iron (IADI) alloyed with different amounts of manganese and nickel was produced. Tensile properties and microstructure were determined. Also, the stability of the austenite during deformation and the lattice strains of the ferrite and the austenite phases were determined using x-ray diffraction (XRD) and neutron diffraction. The results indicated that: 1) high manganese concentrations produced materials with large blocky, low carbon austenite particles at the intercellular boundaries which decreased the elongation of the materials and transformed to martensite during deformation at low strains. 2) Nickel and nickel-manganese alloys formed a combination of blocky and plate-like austenite particles, which were more stable during deformation and either transformed to martensite after yielding or did not transform at all.

Key words: Intercritical austenitizing, intercritical heat treatments, steel, ductile iron, austenite stability, neutron diffraction.
DEDICATION

To my beloved Isabel who fills my life with happiness and love.
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1. INTRODUCTION

Heat treatments are widely used to control the microstructure and hence the mechanical properties of ferrous alloys, which allows an extensive range of applications in different fields and industries. Two of the most common heat treatments performed on ferrous alloys are austempering and quenching and tempering. Both heat treatments start with full austenitization followed by rapid cooling. In quenching, the material is rapidly cooled to a temperature below the martensite finish temperature \( (M_F) \) which causes the transformation of the high temperature austenite to martensite. After quenching, the material is tempered to soften and toughen the hard and brittle as-quenched martensite. The final properties of the material are controlled by the tempering temperature and time.

Austempering is performed by rapidly cooling the material to a temperature above the martensite start temperature \( (M_S) \). This heat treatment cycle leads to the formation of bainite in steels and ausferrite in ductile iron. The final set of mechanical properties of the material is determined by the austempering temperature. In general, the lower the austempering temperature the finer the microstructure, leading to an increase in the strength and a reduction in ductility [1-4].

Intercritical heat treatments are also used in ferrous alloys. In steels, intercritical heat treatments are used to produce DP and TRIP steels with high ultimate tensile strength (UTS), low yield strength (YS) and high elongation to fracture. This combination of properties produces good formability which is a major advantage in the production of sheet products [5-8]. In the last 10 years, intercritical heat treatments have been
successfully applied to ductile iron to produce multiphase matrix structures. Intercritical austenitized ductile irons exhibit a different set of properties than traditional heat treated ductile irons due to the presence of considerable amounts of free ferrite in the microstructure. The microstructures provide ductilities comparable to ferritic ductile iron grades yet higher UTS and YS, which in some cases are comparable with the strengths of regular austempered ductile iron (ADI) and quenched and tempered ductile iron. This newly developed family of ductile iron holds promises for the production of safety components for the automotive and military industries [9-39].

The further development and application of intercritical heat treatments in ferrous alloys require an increasing understanding of different aspects of this kind of heat treatment cycle. For example, intercritical heat treatments have been traditionally applied to low-carbon, low-alloy steels with low hardenability which limits its application to thin sections. The possibility of applying intercritical heat treatments to low alloy common grade steels with higher hardenability can open a new field to produce thicker parts with mechanical properties comparable to those in DP and TRIP steels with potential applications in the casting industry.

In ductile iron, the development of intercritical heat treatments has mainly focused on unalloyed ductile iron with ferritic matrices prior to heat treatment. This practice has left questions about the influence of the as-cast microstructure in the formation of the austenite during the intercritical austenitizing stage and the effect of alloying elements like nickel and manganese on the mechanical properties and the stability of the retained austenite during deformation. Answering these questions can impact positively the understanding and development of this new material.
This dissertation will address in some degree the three aspects previously mentioned by (1) applying intercritical austenitizing followed by austempering to common grade steels, (2) studying the formation of the austenite in the intercritical range in ductile iron alloyed with nickel and manganese and with ferritic-pearlitic microstructures prior to the intercritical heat treatment cycle, and (3) studying the transformation characteristics of the austenite during deformation in intercritically austempered ductile iron alloyed with nickel and manganese using neutron diffraction and X-ray diffraction. The results will open the possibility for new applications of intercritical heat treatments to steels and will bring new knowledge and understanding of intercritical heat treatments in ductile iron. Portions of this research have been previously published and are referenced throughout the text of the dissertation.
2. BACKGROUND AND LITERATURE REVIEW

2.1. Intercritical Heat Treatments in Steels

Intercritical heat treatments in steels start with partially austenitizing the material at the intercritical temperature range, where ferrite and austenite coexist. After intercritical austenitizing, the material can be quenched below the martensite finish temperature to obtain a microstructure of martensite particles in a ferritic matrix. The material can be also quenched after intercritical austenitizing to a temperature above the martensite start temperature and held for a period of time, which as a general rule produces a microstructure of bainite particles in a ferritic matrix [5-8]. This process is called austempering.

The intercritical austenitizing step of the heat treatment plays a major role in the final microstructure of the material and therefore the mechanical properties. For a given alloy chemistry, the volume fraction of austenite is determined by the intercritical austenitizing temperature. It can be deduced from the iron-carbon phase diagram (Figure 1) using the lever rule that increasing the intercritical austenitizing temperature increases the amount of austenite and decreases the amount of ferrite [40]. Therefore, the intercritical austenitizing temperature step determines the amount of austenite transformation products in the final microstructure. In general, increasing the amount of austenite at high temperature (higher intercritical austenitizing temperatures) increases the strength and decreases the ductility of steels with an intercritical heat treatment.

Previous published research of dual phase steels has shown that the formation of the austenite in the intercritical temperature range has three steps [41]. The first step is
the nucleation of the austenite at the ferrite-pearlite grain boundaries and growth of austenite into pearlite. During the second step, austenite grows into the ferrite grains until the equilibrium carbon content and volume fraction is reached. The third step consists of equilibration of the substitutional alloying elements, such as manganese and silicon, between the ferrite and the austenite phases. At temperatures between $A_1$ and 740°C, the first step can take up to 8 hours while the second step can take up to 24 hours. At temperatures above 740°C, austenite growth into pearlite and further growth of austenite into ferrite grains may take less than five minutes. The third step, equilibration of substitutional alloying elements, takes longer because this process is mainly controlled by substitutional diffusion. For example, manganese equilibration between ferrite and austenite can take up to 2000 hours [41, 42]. Therefore, during intercritical austenitizing para-equilibrium conditions (i.e., austenite volume fraction and carbon equilibrium but not substitutional alloying elements equilibrium) rather than equilibrium conditions are achieved [43].

2.1.1. Dual phase (DP) and transformation induced plasticity (TRIP) steels

DP and TRIP steels have high UTS, low YS, high work hardening rate and high ductility. This combination of properties provides good formability which makes these steels a good option for the fabrication of sheet products in the automotive industry. Figure 2 shows the relationship between ultimate tensile strength and elongation for DP and TRIP steels [8].

DP steels are obtained by intercritical austenitizing followed by quenching. Typically, DP steels are produced using low carbon alloys (less than 0.1wt%) with manganese
(1.0-1.5wt%) and silicon (0.6-1.3wt%). Small quantities of molybdenum, vanadium and chromium can be also used as microalloying elements to improve hardenability. The microstructure of DP steels consists of a continuous matrix of ferrite plus particles of martensite (around 20 vol%). The ferrite matrix provides good ductility, and the martensite particles act as the strengthening phase. Small quantities of pearlite, bainite, and retained austenite may also be present in the final microstructure of DP steels depending on the alloy chemistry and quench rate [5].

Typical alloy composition of TRIP steels are 0.1-0.4% wt carbon, 1.5 wt% manganese and 1.5 wt% silicon. These steels may also contain small quantities of titanium, niobium and vanadium. TRIP steels are produced by intercritically austenitizing followed by austempering [8]. The intercritical austenitizing step of the heat treatment determines the amount and carbon content of the austenite at high temperature, while the austempering step determines the morphology of the bainitic product. The austempering temperature lies between the temperature to form pearlite and the martensite start temperature. In general, a high austempering temperature leads to the formation of upper bainite, which consists of lath or plate-shaped ferrite arranged in packets and interlath carbide precipitates. At lower austempering temperatures, the austenite transformation product is lower bainite, which consists of fine ferrite needles plus fine carbides. The transition temperature from lower to upper bainite depends on the chemistry of the austenite particles. Lower bainite is stronger and less ductile than upper bainite [44]. In TRIP steels, silicon plays an important role because it delays the formation of carbides during the bainitic transformation leading to the presence of retained austenite at ambient temperature. The retained austenite improves the ductility and the strain hardening coefficient of
the material by an effect known as transformation induced plasticity, which consists of the transformation of the retained austenite to martensite during plastic deformation [45, 46].

DP and TRIP steels have low hardenability because of their low carbon and low alloying concentration. For example, Topcu et al. studied the hardenability of a 0.28C, 1.45Mn, 0.20Si, 0.21Cr, 0.13V, 0.01Nb steel intercritical austenitized between 737-779°C for 30 minutes. The hardenability was determined using a Jominy test. The results showed that the maximum achievable hardness depended on the volume fraction of martensite in the microstructure and that the effective hardness depth of the steel investigated was 5 mm [47].

2.1.2. Intercritical heat treatments in common grade steels

The literature related to intercritical heat treatments in common steel grades is limited. The development of intercritical heat treatments for common grade steels would not require developing new alloys. In addition, the higher alloying content in common steel grades would allow for the use of materials with a higher hardenability that could be used to produce thicker section parts than those obtained with DP and TRIP steels. These may have potential application in the casting industry.

Bakhtiari et al. investigated the effect of the austempering temperature in a 4340 steel after intercritical austenitizing. The heat treatment cycle consisted of fully austenitizing at 850°C for one hour followed by heating at 700°C for 90 minutes. The material was then austempered at 300-450°C. This heat treatment cycle provided a microstructure of ferrite plus bainite particles. The results showed that strength decreased as austemper-
ing temperature increased from 300°C to 400°C, which was attributed to transition from lower to upper bainite. Also, strength increased as the austempering temperature increased from 400°C to 450°C due to incomplete transformation of austenite to bainite and further transformation of austenite to martensite upon cooling to room temperature [44].

Previous published work indicated that intercritically austenitizing followed by tempering on common grade steels 8620, 4330 and 4340 produces materials with multiphase microstructures and tensile properties comparable to those in DP and TRIP steels [16].

2.2. *Intercritical Heat Treatments in Ductile Iron*

Intercritical heat treatments in ductile iron start in the same fashion as in steel. The iron is first intercritically austenitized. After that, the material can be quenched or austempered. In the first case, the material must also be tempered in order to soften and toughen the hard and brittle as-quenched martensite. For a given ductile iron chemistry, the amount of austenite at high temperature is determined by the intercritical austenitizing temperature. Also, it has been reported that at the intercritical austenitizing temperature the austenite starts to nucleate at the eutectic cells and the intercellular boundaries, which are considered the preferential sites for heterogeneous nucleation [10,12,17-24].

The carbon content of the high temperature austenite is determined by the intercritical austenitizing temperature. Studies performed in unalloyed ductile iron with ferritic matrices prior to heat treatment have shown that the carbon to form the austenite at high temperature comes from the graphite nodules; and, therefore, the carbon concentration in the austenite increases as the intercritical austenitizing temperature increases [21-22]. Some authors have used ductile iron with ferritic-pearlitic matrices as starting mi-
crostructures [12-15, 28-36]. Druschitz et al. suggested that under this condition the carbon to form the austenite at high temperature comes from the carbon in the pearlite and the diffusion of carbon from the graphite nodules is limited [12]. When the pearlite is completely dissolved the graphite nodules start providing carbon to reach the equilibrium austenite volume fraction and carbon concentration. Figure 3 shows the intercritical regions in the iron-carbon phase diagram with 2% silicon. Region 1 corresponds to the intercritical austenitizing region for ductile iron with an initial fully ferritic matrix, while Region 2 is the presumed intercritical region for ductile iron with an initial ferrite-pearlite matrix.

There is no available data in the literature about the segregation of substitutional elements in ductile iron during intercritical austenitizing. However, studies performed in DP steels suggest that the partitioning of elements such as silicon, nickel and manganese during intercritical austenitizing may be limited due to the low rate of substitutional diffusion at the temperatures of the heat treatment [41]. As a result, the concentration of those elements in the austenite is determined mainly by the chemistry of the alloy and their segregation during the solidification process. Therefore, austenite particles formed at the eutectic cells are expected to have a higher manganese concentration. While austenite particles formed near the graphite nodules will have a higher concentration of nickel, silicon and copper.

2.2.1. Intercritical quenched and tempered ductile iron (IAQ&TDI)

IAQ&TDI, also known as dual phase ductile iron, is obtained by intercritical austenitizing followed by quenching and tempering, which provides a microstructure of
graphite nodules in a matrix of ferrite plus tempered martensite particles. The matrix of the iron resembles the microstructure of dual phase steels [9-12]. The main difference in the intercritical heat treatment of DP steels and IAQ&TDI is that the former is not usually tempered after quenching, while the latter is usually tempered after quenching. Tempering consists of heating the material below the eutectoid temperature which leads to the decomposition of the untempered martensite into ferrite plus iron carbides precipitates. In general, tempering decreases strength and hardness and increases ductility and toughness [48, 49]. Potential applications of IAQ&TDI include suspension components which require good toughness and ductility. The strength depends strongly on the volume fraction of martensite in the microstructure, while the elongation is mainly related to the tempering conditions. Typical tensile properties of IAQ&TDI are 600-800 MPa UTS, 400-450 MPa YS and 5-12% elongation [10-11].

Research has been performed in order to elucidate the role of tempering conditions and martensite volume percent and morphology on the mechanical properties of IAQ&TDI. These studies have used unalloyed ductile iron and the matrices prior to heat treatment have been fully ferritic. Cerah et al. produced ferrite-martensite matrices in ductile iron by intercritically austenitizing at 795°C-815°C for 20 minutes followed by quenching in oil at 100°C. After that, the material was tempered at 500°C for 1h-5h. The results indicated that the variation of strength and ductility with tempering time were related to the intercritical austenitizing temperature, which controlled the carbon content of the martensite [11].

Rashidi et al. investigated the effect of tempering conditions in ductile iron having a ferritic-martensitic microstructure. The material was austenitized at 900°C for 480 se-
conds and then quenched in water. The results indicated that impact strength and elongation increased when tempering was performed at 400°C-500°C for 60-120 minutes, whereas ultimate tensile strength and yield strength remained almost unchanged [9]. An austenitization temperature of 900°C is above the intercritical temperature range so it can be inferred that incomplete austenitizing rather than intercritical austenitizing was used for the authors to obtain the desired microstructure. The amount of austenite at high temperature, and hence the amount of martensite in the final microstructure, is more difficult to control using this method than using intercritical austenitizing. Also by this approach, the high temperature austenite nucleates mainly in the areas surrounding the graphite nodules where carbon is easily available rather than in the eutectic cells [27].

Kocatepe et al studied the effect of martensite volume fraction and its morphology on the tensile properties of ductile iron with a ferritic-martensitic matrix. The material was austenitized between 780°C and 840°C for 30 seconds and then quenched in oil at 100°C. After that, the samples were tempered at 550°C for 1-3 hours. The results showed that ferritic-martensitic microstructures in ductile iron provided higher elongation than fully martensitic microstructures. Also, strength increased and elongation decreased as the volume fraction of martensite increased. Strength increased as martensite particle size decreased [10].

2.2.2. Intercritical austempered ductile iron (IADI)

IADI also known as dual phase austempered ductile iron is obtained by intercritically austenitizing followed by austempering, which produces a microstructure of austenite particles in ferritic matrix. The austenite particles can be present between bainitic fer-
rite (ausferrite) or as untransformed austenite volumes. IADI is a viable alternative to produce powertrain and chassis components for the automotive and military industry, since it possesses high YS (~380-550 MPa), high UTS (~500-900 MPa), high elongation to fracture (~14%-20), good fracture toughness (~45-55 MPa-m$^{1/2}$), good resistance to environmental cracking, excellent castability and excellent machinability [13-15, 17-39].

IADIs have different stress-strain characteristics than ferritic ductile irons, pearlitic ductile irons and conventional austempered ductile irons. Figure 4 compares the relationship between UTS and elongation for ductile iron with different microstructures including IADI [50]. Compared to ferritic ductile iron, intercritically austempered ductile iron has nearly twice the YS yet similar high elongation-to-fracture. Compared to pearlitic ductile iron, intercritically austempered ductile iron has slightly higher YS and more than double the elongation to fracture. Compared to conventional austempered ductile iron (ADI), intercritically austempered ductile iron has lower YS but nearly double the elongation to fracture [15].

As in other intercritical ferrous materials, the intercritical austenitizing step of the heat treatment has a large effect on the mechanical properties because it controls the amount of free ferrite and austenite products in the final microstructure. The austempering step of the heat treatment has a lesser effect on the mechanical properties of IADI than conventional ADI [19, 28-29]. However, it is worthwhile to explain the mechanism of the bainitic reaction in ductile iron based on studies performed with regular ADI.

Conventional ADI is produced by fully austenitizing followed by austempering. During full austenitization, the microstructure consists of graphite nodules in an austenitic matrix. The material is then austempered, which consists of quenching in a salt bath to
a temperature between the temperature of pearlite formation and the martensite start temperature. The bainitic reaction that occurs during the austempering step of the heat treatment in ductile iron has two stages. The first stage, also known as the processing window, consists of the nucleation and growth of ferrite. The low solubility of carbon in ferrite causes carbon to be rejected to the austenite phase and the high silicon concentration delays the precipitation of carbides. When the material is halted at the end of this stage, the austenite can have a concentration of carbon high enough to be stable upon quenching to room temperature, which leads to the formation of an aggregate of ferrite and stabilized austenite known as ausferrite. It is at the end of the first stage that the best mechanical properties of the material are achieved. The second stage of austempering starts with the precipitation of carbides followed by growth of ferrite plates at the expense of the austenite phase [51-54].

The morphology of the ausferrite depends on the austempering temperature. At high austempering temperature, the driving force for the reaction is low and the diffusion of carbon is high leading to the formation of low amounts of ferritic bainite and large volume fractions of austenite. At low temperatures, the driving force for the reaction is high and carbon diffusion is slow and then more and finer ferritic bainite is formed [54].

As was mentioned before, silicon delays the precipitation of carbides allowing the existence of a processing window for the production of ADI. Alloving elements such as manganese and nickel also play an important role in the production of regular ADI. Manganese is an austenite stabilizer and a powerful carbide former which segregates preferentially to the intercellular boundaries during solidification. Manganese also retards the bainitic reaction leading to the formation of low carbon, untransformed austenite vol-
umes at the intercellular boundaries, which reduce the ductility of the material [52, 53]. Because of this, in conventional ADI, the concentration of manganese is limited in order to avoid its negative effects. Nickel is an austenite stabilizer that also increases hardenability of ADI and reduces the tendency for carbide formation [55].

The first known attempt to produce ferritic-ausferritic matrices in ductile iron was made by Wade et al. [56]. The microstructure was accomplished by quick and incomplete austenitization at temperatures in the fully austenitic field. The amount of austenite at high temperature depended on the austenitization time, i.e., longer times provided larger volume fractions of austenite. Also, the austenite nucleated and grew mainly in the areas surrounding the graphite nodules [27].

Kobayashi et al. used a different approach than Wade. In this case, the duplex microstructures were obtained by intercritical austenitizing. The authors tried to preferentially introduce an ausferritic phase in the areas surrounding the graphite nodules and the intercellular boundaries (which correspond to the sites of crack initiation in ductile iron) while keeping an interconnected matrix of ferrite, which led to the development of a material tougher than regular ADI [57]. Aranzabal et al. produced ferrite-ausferrite matrices in ductile iron by intercritically austenitizing followed by austempering. The variation in the amount of ausferrite was accomplished by using different silicon concentrations in the alloys under study and keeping the intercritical austenitizing temperature constant. The results suggested that ductile iron with a ferrite-ausferrite matrix exhibited strength comparable to pearlitic microstructures, while ductility was comparable with ferritic grades [17-18].
Druschitz et al. used intercritical austenitizing followed by austempering at 315°-400°C of ductile iron alloyed with nickel (~1.85 wt%), copper (~0.85 wt%) and molybdenum (~0.05 wt%) with a ferrite-pearlite microstructure prior to heat treatment to produce ferritic-austenitic microstructures. The authors found that (1) hardness increased as the intercritical austenitizing temperature increased, (2) hardness and yield strength were linearly related, i.e., as hardness increased yield strength increased, (3) the austempering temperature had a mild effect on the mechanical properties, which were mainly controlled by the intercritical austenitizing step of the heat treatment, and (4) intercritically austenitizing followed by austempering produced materials with a much better machinability, fatigue life and resistance to environmental cracking than regular ADI [28-36].

Kilicli et al. conducted detailed research on the influence of the intercritical austenitizing temperature and austempering time on the microstructure and tensile properties of unalloyed ductile iron austempered from the intercritical region. The authors used a ferritic matrix prior to heat treatment to perform the study. They showed that increasing the intercritical austenitizing temperature increased the amount of austenite products and decreased the amount of free ferrite in the final microstructure as described by the phase diagram and the lever rule. The results also showed that increasing the intercritical austenitizing temperature increased the carbon content in the austenite. The materials exhibited higher ductility than regular ADI and strength comparable to pearlitic ductile iron [21].

Basso et al. used unalloyed ductile iron with a ferritic matrix prior to heat treatment. The materials were intercritically austenitized at temperatures between 780 and 860°C followed by austempering at 300, 330 and 350°C to determine the influence of the
austenitizing and austempering temperatures on the microstructure, tensile properties and fracture toughness. The results revealed that the mechanical properties were determined mainly by the intercritical austenitizing step of the heat treatment, while the austempering step had a mild effect. The effect of the austempering temperature on properties increased as the amount of ausferrite in the final matrix increased. The best toughness was obtained for materials with a volume percent of ausferrite around 30% [19,20].

Erdogan et al. studied the influence of austenite dispersion on the phase transformation during austempering of intercritically austenitized ductile iron. The difference in austenite dispersion between samples was accomplished by using two different microstructures prior to heat treatment: (1) fully martensitic and (2) fully ferritic. The results showed that the former provided a finer and more dispersed ausferrite than the latter because of more sites for heterogeneous nucleation during the austempering process. Also, a finer microstructure produced a larger volume fraction of high carbon austenite, stabilized the austenite carbon content much earlier and widened the processing window [22,26].

2.3. Austenite Stability during Deformation

The stability of the austenite during deformation can play an important role in the mechanical properties of ferrous materials increasing the strain hardening rate and the elongation by an effect called transformation induced plasticity. During transformation induced plasticity, the austenite transforms to martensite under strain or stress with a subsequent change in volume and shape of the transformed region. Carbon concentration, morphology, and size are the main factors that determine the austenite stability during deformation. In general, as the carbon concentration increases, the stability of austenite
increases. It has been reported that low carbon austenite (<0.05-0.06 wt%) transforms to martensite rapidly and does not contribute to an increase in elongation, while the transformation of high carbon austenite (>1.8 wt%) is limited. Also, large austenite particles are less stable than small austenite particles. In general, it can be said that austenite particles larger than 1μm transforms to martensite at very low strains and austenite particles smaller than submicron size have a low tendency to transform to martensite. Regarding austenite morphology, blocky austenite tends to transform easily during deformation at very low strains, while film austenite particles within the subunits of bainitic ferrite does not transform to martensite during deformation [58,59].

The stability of austenite has been studied in TRIP steels, multiphase steels and austenitic stainless steels. Different methods and characterization techniques have been used to develop and understand the role of the austenite on the mechanical properties. Among these techniques, neutron diffraction has proven to be an effective way to study the stability of austenite during deformation and to determine the elastoplastic properties of ferrous materials [60-65]. Neutrons have a greater penetration depth than x-rays (more than 10 mm for steel), while x-rays only provide information from the near surface (10’s of μm) [14]. The diffraction elastic constants of the phases in the material can also be determined with neutron diffraction but cannot be obtained with x-ray diffraction [66].

Using neutron diffraction, it was determined that the elastoplastic properties of the phases in multiphase steels are related to the thermomechanical processing conditions and that the rate of transformation of the austenite to martensite during deformation did not depend upon the amount of initial retained austenite in the material [60-62]. Jacques et al. studied multiphase steels during tensile loading and found that the YS of the austen-
ite increased as the carbon content increased [63]. Oliver et al. studied the stress induced martensitic transformation in TRIP steels and found that the austenite grains having the \langle 100 \rangle parallel to axial load transformed preferentially. They also found that the load was transferred from the original austenite to the evolving martensite during plastic deformation [64].

Studies of the stability of the austenite during deformation in ductile iron processed from the intercritical range are limited. However, some understanding can be inferred from studies performed on regular ADI. In ADI, the formation of strain induced martensite was found to depend upon the austenitizing and austempering temperatures. High austenitizing and austempering temperatures favor the formation of low carbon blocky austenite, which transformed to martensite during deformation at small strains. On the other hand, low austenitizing and austempering temperatures produce small austenite particles with high carbon concentration that were more stable [67].
Figure 1. Schematic portion of the Fe-C phase diagram.

Figure 2. Relationship between ultimate tensile strength and elongation of DP and TRIP steels [8].
Figure 3. Schematic portion of the Fe-C 2%Si phase diagram showing the intercritical austenitizing regions for ductile iron with fully ferritic and ferritic-pearlitic matrices before intercritical heat treatment.

Figure 4. Relationship between elongation and tensile strength for ductile iron with different microstructures [50].
3. SPECIFIC AIMS

The research objective of this dissertation was to study intercritical heat treatments in ductile iron and steel. Materials such as intercritically austempered ductile iron (IADI), dual phase (DP) and transformation induced plasticity steels (TRIP) are produced by the proper combination of intercritical heat treatment and alloy chemistry. These materials can provide significant weight savings in the automotive industry. Studying intercritical heat treatments allows a better understanding of the relationship between mechanical properties, heat treatment conditions and alloy chemistry. This knowledge can be further used to optimize the production parameters of intercritically heat treated materials and enhance the applications of intercritical heat treatments. Specifically, the following three objectives were addressed.

3.1. **Aim 1: Determine the relationship between heat treatment conditions, alloy chemistry and tensile properties in common grade steels with an intercritical austempered heat treatment.**

Intercritical heat treatments have been traditionally applied to low carbon-low alloy steels with low hardenability which limits its application to thin sections. The possibility of applying intercritical heat treatments to common grade steels with higher hardenability can open a new field to produce thicker parts with mechanical properties comparable to those in DP and TRIP steels with potential applications in the casting industry. A previous study indicated that intercritical austempered heat treatment applied to common grade steels (wrought steels AISI 4340, 4330 and 8620 and a cast steel) produced
materials with tensile properties in the same range of the tensile properties of DP and TRIP steels [16]. However, there is no certainty about the final microstructures and tensile properties that could be obtained and there is no understanding of the relationship between tensile properties, alloy chemistry and heat treatment conditions.

3.2. **Aim 2: Determine the effect of heat treatment conditions on the tensile properties and microstructure in Intercritically Austenitized Quenched and Tempered Ductile Iron (IAQ&TDI).**

The development of intercritical heat treatments in ductile iron has focused mainly on unalloyed ductile iron with ferritic matrices prior to heat treatment. This has left questions about the influence of the as-cast microstructure in the formation of the austenite during the intercritical austenitizing stage and the effect of alloying elements like nickel and manganese in the mechanical properties of IAQ&TDI. Successful completion of this objective might enhance the potential applications of intercritical heat treatments in ductile iron.

3.3. **Aim 3: Determine the effect of manganese and nickel on the tensile properties, stability of the austenite under load conditions and lattice strain of the austenite and ferrite phases in Intercritically Austempered Ductile Iron (IADI).**

The mechanical properties of IADI are determined by the volume fraction and the mechanical properties of the matrix phases, ferrite and austenite, which are determined by the chemistry of the alloy and the heat treatment conditions. There is no quantitative data about the effect of manganese and nickel on the mechanical properties of IADI. Also, there is no understanding of the effect of manganese and nickel on the mechanical properties of the matrix phases and the stability of the austenite phase under load conditions.
Understanding the effect of manganese and nickel on the mechanical properties of the matrix phases in IADI will allow further optimization of alloy chemistry and heat treatment conditions to achieve the best combination of mechanical properties.
4. MATERIALS AND METHODS

4.1. General Methods

4.1.1. Production of the ductile iron alloys

Around ~70 pound heats of each ductile iron alloy were produced at the casting laboratory at the University of Alabama at Birmingham (UAB) using an induction furnace. Low carbon steel punching, granular silicon carbide, granular carbon riser, ferro-manganese, copper turnings and nickel shot were used to prepare the alloys. The ductile iron treatments were performed in an open ladle using the sandwich method. Chemistry samples of the treated iron were taken. The chemistry was determined by optical emission spectroscopy (OES) of chilled samples. Carbon and sulfur were determined by combustion analyses using a Leco C/S analyzer. The treated iron was poured in Y-blocks made from chemical bonded sand using 1.5% binder (Figure 5). The castings were shaken out ~1 hour after pouring.

4.1.2. Determination of the intercritical austenitizing temperatures

Samples of the alloys were placed in a pre-heated laboratory furnace, held 2.5-4 hours at temperature and quenched in water to transform the austenite present to martensite. The alloys were tempered at 150°C-200°C for one hour to facilitate metallographic examination. Samples were sectioned, mounted and polished using standard metallographic techniques and the percent ferrite and martensite (austenite during heat treatment)
were determined by image analysis. For image analysis, twenty randomly selected fields of view were examined at a magnification of 500X.

4.1.3. Heat treatments

The intercritical austenitizing temperatures and times were selected according to the results of the previous stage of the study (Section 4.1.2). Intercritically austempered heat treatments for steel and ductile iron samples were performed at a commercial heat treat facility (Objectives 1 and 3). Samples were intercritically austenitized and then austempered in a neutral salt bath. After austempering, the samples were allowed to cool for 10-15 minutes and then quenched in water to minimize the formation of residual stresses and to clean off the salt. Austempering conditions were 300-400°C for 10 minutes for commercially available steels and 357°C for 2.25 hours for IADI.

Intercritically quenched and tempered heat treatments were performed at UAB. Samples were intercritically austenitized in a pre-heated laboratory furnace and then quenched in a polymeric solution UCON E 20% at ambient temperature. Three intercritical austenitizing temperatures were used to obtain different amounts of martensite. Also, three different tempering conditions were evaluated: untempered, tempered at 400°C for one hour and tempered at 500°C for one hour. The results obtained with the untempered samples were used as a baseline.

4.1.4. Microstructure characterization

Samples of the heat treated materials were sectioned, mounted and polished using standard metallographic techniques with a 1µm diamond final polish. The samples were etched with 3% nital. The microstructures were observed and digital images taken using
optical microscopy. An FEI FEG 650 scanning electron microscope (SEM) equipped with an EDAX TEAM energy dispersive x-ray system (EDS) was also used to characterize the microstructure of the materials for Objectives 1 and 3.

4.1.5. Tensile properties and hardness

Tensile bars, 9 mm diameter x 36 mm gage length, were machined from the heat treated materials and tested in accordance to ASTM E8 on an MTS Model 810 servo hydraulic tension/compression tester [68]. Ultimate tensile strength (UTS), yield strength (YS) and elongation to fracture were determined. Three tensile specimens for each alloy heat treatment combination in the steels and two tensile specimens for each alloy heat treatment combination for the ductile irons were tested to determine the tensile properties.

Brinell hardness was determined on the ductile iron samples (Objectives 2 and 3) using a NewAge model HB3000B hardness tester with a Brinell Optical Scanning System (B.O.S.S.) model #OS100 optical reader and Computer Assisted Measurement System (C.A.M.S.) software. A 10 mm diameter steel ball and a load of 3000 kg were used for the Brinell hardness measurements.

4.2. Materials and Methods – Aim 1: Intercritically Austempered Steel

4.2.1. Materials

Three wrought commercial available steels 8620, 4130 and 4340 and one cast steel were used for this part of the study. The cast steel was provided by a steelmaking company. Table 1 lists the chemical composition of the alloys. The intercritically austenitizing temperatures were determined experimentally (Section 4.1.2). Next the materials
were intercritically austempered (Section 4.1.3.). Microstructure, tensile properties and hardness were determined (Sections 4.1.4 and 4.1.5).

**4.2.2. Statistical analyses of the information**

Statistical multi linear regression analyses were used to determine the effect of alloy chemistry and heat treatment conditions on the volume percent of austenite at high temperature and the tensile properties. Statistical analysis was accomplished using MINITAB [66]. Only first and second order interactions with \( p \) values < 0.05 were taken into account for the multi linear regression models.

**4.3. Materials and Methods – Aim 2: Intercritically Quenched and Tempered Ductile Iron**

**4.3.1. Materials**

A ductile iron alloyed with manganese and nickel was produced for this part of the study (Section 4.1.1.). Table 2 lists the chemistry of the alloy. The intercritical austenitizing temperatures were determined experimentally (Section 4.1.2.). The materials were heat treated and microstructure, tensile properties and hardness were determined (Sections 4.1.3, 4.1.4 and 4.1.5). The volume fraction of martensite was determined by image analyses. Martensite and ferrite microhardness were determined using a Buehler Micromet II Microhardness tester. Ten indentations were done in each sample with a load of 25 grams. Semi-quantitative compositions of martensite and ferrite were obtained using EDS with SEM at a 30 kV accelerating voltage. The analyses were performed on the untempered samples. Sixteen analyses were performed in each phase per sample. XRD was accomplished using a Siemens D-500 Diffractometer with Cu radiation. The scans were
performed for 20 between 40° and 90° with a step size of 0.02° and a dwell time of 5 seconds. The results were used to determine if some retained austenite was present in the untempered samples.

4.4. Materials and Methods – Aim 3: Intercritically Austempered Ductile Iron

4.4.1. Materials

The alloys were prepared in accordance with the procedure described in Section 3.1.1. Four different ductile iron alloys were used for this part of the study: low nickel, high nickel, high manganese and manganese-nickel. Table 3 lists the chemistry of the alloys. The intercritical austenitizing temperatures were determined experimentally (Section 4.1.2.). Next, the materials were intercritically austempered (Section 4.1.3.). Microstructure, tensile properties and Brinell hardness were determined for the heat treated samples (Sections 4.1.4 and 4.1.5). Neutron diffraction and XRD were used to determine the stability of the austenite phase under load conditions. Lattice strains of the ferrite and austenite phases and specific Diffraction Elastic Constants (DEC\textsubscript{hkl}) were determined as well.

4.4.2. X-ray diffraction (XRD)

After tensile testing, samples for XRD were taken from the grip and from the gage (close to the fracture surface) of the regular tensile specimens and analyzed in the directions perpendicular and parallel to the load axis. The samples taken from the grip were considered unloaded/unstrained and the samples taken from the gage were considered loaded/strained. The samples were sectioned with a diamond saw and polished using standard metallographic techniques with a 1µm diamond powder final polish. XRD were
performed using a Siemens D-500 Diffractometer with Cu radiation and a graphite monochromater. The scans were performed at 40kV and 30 mA for 2θ between 20° and 90° with a step size of 0.04° and a dwell time of 30 seconds. XRD results were used to determine austenite volume percent before and after loading and austenite lattice parameter. Quantitative analyses were conducted using JADE wherein the two theta zero, background, scale, lattice parameter, preferred orientation, peak shape, U (peak sharpness) and W (peak width) were refined [70]. The space group, atomic positions and starting cells for BCC and FCC iron phases and graphite were obtained from the PDF database (04-002-1253, 00-052-0512 and 00-056-0160, respectively) [71]. Carbon content in the austenite was estimated from the lattice parameter using Equation 1 which provides an approximate value of the carbon content in the austenite that was used for comparison between samples [72].

\[ \%C = \left( a_\gamma - 3.555 \right)/0.044 \]  
Equation 1

4.4.3. Neutron diffraction

Special tensile samples, 4 mm diameter x 25 mm gage length, were machined from the IADI samples to perform the neutron diffraction experiments. The studies were performed using a load frame at the second generation neutron residual stress load facility at HFIR (High Flux Isotope Reactor) at Oak Ridge National Laboratory (Figure 6).

Axial load was incrementally increased and diffraction data (lattice spacing and intensity area) obtained parallel or perpendicular to the loading axis at each load level. The information was collected for the ferrite 110 and 200 reflections and for the austenite
111 and 311 reflections. This allowed the in-situ determination of the lattice strain of the individual phases (ferrite and austenite). Lattice strains were calculated using Equation 2.

\[
Lattice\ strain, \ ppm = \frac{(d_{hkl} - d_{hkl}^0)}{d_{hkl}^0} \times 10^6 \quad \text{Equation 2}
\]

where \(d_{hkl}\) is the lattice spacing of a particular plane when loaded and \(d_{hkl}^0\) is the lattice spacing of that plane at no load.

Determination of the stability of the austenite phase under load conditions was calculated. A measure of the amount of austenite at each load was calculated from the integrated intensities areas of the reflections FCC111, FCC311, BCC110 and BCC200 using Equation 3. The amount of austenite remaining (\(\Delta\)) at each applied load compared to that present at no load was determined using Equation 4.

\[
\gamma = \frac{(\text{FCC111} + \text{FCC311})}{(\text{FCC111} + \text{FCC311}) + (\text{BCC110} + \text{BCC200})} \times 100 \quad \text{Equation 3}
\]

\[
\Delta = \frac{\gamma_{\text{load}}}{\gamma_{\text{no load}}} \times 100 \quad \text{Equation 4}
\]
Table 1. Chemical composition of steels investigated

<table>
<thead>
<tr>
<th>Material</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>S</th>
<th>P</th>
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<td>0.025</td>
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<td>0.74</td>
<td>0.26</td>
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<td>0.83</td>
<td>0.20</td>
<td>0.009</td>
<td>0.007</td>
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<tr>
<td>Cast steel</td>
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<td>0.88</td>
<td>0.47</td>
<td>0.69</td>
<td>0.80</td>
<td>0.43</td>
<td>0.016</td>
<td>0.014</td>
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Table 2. Chemical composition of IAQ&TDI investigated

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<tr>
<th>Element, wt%</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
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<th>Cu</th>
<th>Mg</th>
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<td>0.05</td>
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Table 3. Chemical composition of IADI investigated

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<td>2.17</td>
<td>0.33</td>
<td>0.68</td>
<td>0.72</td>
<td>0.013</td>
<td>0.045</td>
<td>0.013</td>
</tr>
<tr>
<td>High Ni</td>
<td>3.92</td>
<td>2.34</td>
<td>0.33</td>
<td>2.47</td>
<td>0.75</td>
<td>0.012</td>
<td>0.040</td>
<td>0.016</td>
</tr>
<tr>
<td>High Mn</td>
<td>3.73</td>
<td>2.25</td>
<td>1.59</td>
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<td>0.04</td>
<td>0.017</td>
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<tr>
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<td>0.05</td>
<td>0.016</td>
<td>0.048</td>
<td>0.014</td>
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</table>

Figure 5. Dimensions of the Y-Block used to produce the ductile iron castings.
Figure 6. (a) Load frame at the 2\textsuperscript{nd} generation neutron residual stress facility (NRSF2) at the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory. (b) Close-up of grips, tensile specimen, extensometer, incident beam collimator and diffracted beam collimator.
5. INTERCRITICALLY AUSTEMPERED STEEL

by

RICARDO E. ARISTIZABAL, ROBIN D. FOLEY, ALAN P. DRUSCHITZ AND MEL OSTRANDER

In preparation for AFS Transaction

Format adapted for dissertation
Abstract

Intercritical heat treatments are commonly used in steels to produce materials such as dual phase (DP) and transformation induced plasticity steels (TRIP) with applications in automotive sheet products. In this study, the intercritical austenitizing temperatures for different steel grades (8620, 4330 and 4340) and a cast steel were experimentally determined. The steels were intercritically austenitized followed by austempering (intercritically austempered) over a range of processing condition. This paper reports the microstructure and mechanical properties of the materials. The results showed that applying intercritical austempered heat treatments to common grade steels produced multi-phase microstructures and tensile properties comparable to those of dual phase steels and transformation induced plasticity steels.

Key words: Intercritically austenitizing, austempering, steel.
Introduction

Intercritical heat treatments are used in steels to produce multiphase microstructures, such as the microstructure of dual phase (DP) and transformation induced plasticity steels (TRIP). These steels have a unique combination of mechanical properties, i.e., low yield strength, high ultimate tensile strength and high elongation to fracture, which make them suitable for automotive sheet products [1-4].

Intercritical heat treatments start with intercritical austenitizing, or intercritical annealing, which consists of partially austenitizing the material at the intercritical temperature range, where ferrite and austenite coexist. The formation of austenite in the intercritical temperature range has three stages [5]. The first step is the nucleation of the austenite at the ferrite-pearlite grain boundaries and growth of austenite into pearlite. During the second step, austenite grows into the ferrite grains until the equilibrium carbon concentration and volume fraction is reached. The third step consists of equilibration of the substitutional alloying elements, such as manganese and silicon, between the ferrite and the austenite phases. At temperatures between \( A_1 \) and 740°C, the first step can take up to 8 hours while the second step can take up to 24 hours. At temperatures above 740°C, austenite growth into pearlite and further growth of austenite into ferrite grains may take less than five minutes. Times for equilibration of substitutional alloying elements are longer because this process is mainly controlled by substitutional diffusion of atoms. For example, manganese equilibration between ferrite and austenite can take up to 2000 hours [5, 6]. Therefore, during intercritical austenitizing para-equilibrium conditions (i.e., austenite volume fraction and carbon equilibrium but not substitutional alloying elements equilibrium) rather than equilibrium conditions are achieved [7].
After intercritical austenitizing, the material can be fast cooled to a temperature below the martensite start temperature (quenching), transforming the high temperature austenite to martensite. Intercritical austenitizing followed by quenching is used to produce DP steels. Typically, DP steels are produced using low carbon alloys (less than 0.1wt%) with manganese (1.0-1.5wt%) and silicon (0.6-1.3wt%). Small quantities of molybdenum, vanadium and chromium can be also used as microalloying elements to improve hardenability [1]. The microstructure of DP steels consists of a continuous matrix of ferrite plus particles of martensite (around 20 vol%). The ferrite matrix provides good ductility and the martensite particles act as the strengthening phase. Small quantities of pearlite, bainite and retained austenite may also be present in the final microstructure of DP steels depending on alloy chemistry and cooling rate during quenching.

After intercritical austenitizing the material can also be fast cooled to a temperature above the martensite start temperature (austempering). This process leads to the transformation of austenite to bainite, which can be upper or lower bainite depending on the austempering temperature, i.e., high austempering temperatures lead to the formation of upper bainite while low transformation temperatures produce lower bainite [8]. Intercritical austempering is used to produce TRIP steels [3]. Typical compositions of TRIP steels are 0.1-0.4%wt carbon, 1.5 wt% manganese and 1.5 wt% silicon. It may also contain small quantities of titanium, niobium and vanadium. Silicon retards the formation of carbides during the bainitic transformation leading to the presence of retained austenite at ambient temperature, which improves the ductility of the material [9, 10]. Bakhtiari et al investigated the effect of austempering temperature in a 4340 steel after intercritical austenitizing. The results showed that strength decreased as austempering temperature in-
creased from 300°C to 400°C, which was attributed to transition from lower to upper bainite. Also, strength increased as the austempering temperature increased from 400°C to 450°C due to incomplete transformation of austenite to bainite and further transformation of austenite to martensite upon cooling to room temperature [8].

In the present study, the intercritical austenitizing temperatures for common steel grade bars 8620, 4330 and 4340 and a cast steel were experimentally determined. Then, a series of intercritical austempering heat treatments over a variety of processing conditions were performed. This paper reports the microstructure and tensile properties obtained.

**Experimental**

**Materials**

Three commercially available wrought steels and one cast steel were examined in this study. 8620, 4330 and 4340 steels were obtained as 16 mm diameter cold finished round bars. The chemical compositions were determined by Optical Emission (arc-spark) Spectrometry (OES) (Table 1).

**Determination of the intercritical austenitizing temperatures**

Samples of the alloys were placed in a pre-heated laboratory furnace, held 3 hours at temperature and then quenched in water to transform any austenite to martensite. After that, the samples were tempered at 150°C for one hour to facilitate metallographic examination. Samples were sectioned, polished using standard metallographic techniques and the percent martensite (austenite during heat treatment) were determined by image analysis. For image analysis, twenty randomly selected fields of view were examined at a magnification of 500X. Linear regression analysis was performed in order to correlate
the effect of chemistry and austenitizing temperature on the percent martensite (austenite during heat treatment). The information was used to determine the appropriate intercritical austenitizing temperatures to obtain a matrix of interconnected ferrite. The intercritical austenitizing temperatures for the next stage of the study were selected to obtain 30-40 vol% of austenite at high temperature.

**Heat treatment**

Round bars 16 mm diameter, 150 mm long for the wrought steels and square bars 16 mm x 16 mm, 110 mm long for the cast steel were intercritically austenitized for 3 hours in a nitrogen atmosphere and then austempered in a neutral salt bath at a commercial heat treat facility. Two different intercritical asutenitizing temperatures were used for the steels. Austempering treatments ranging from 300°C to 400°C for 10 minutes were used for the steels. Table 2 lists the austempering temperatures used. After austempering, the samples were allowed to cool for 10-15 minutes and then quenched in water to minimize the formation of residual stresses and to clean off the salt. The microstructures of the heat treated samples were determined using an optical microscope and a scanning electron microscope (SEM). Samples were prepared using standard metallographic techniques. Tensile specimens, 9 mm diameter x 36 mm gage length, were machined from the heat treated bars and tested in accordance to ASTM E8-04 on a MTS Model 810 servo hydraulic tension/compression machine, which allowed the ultimate tensile strength (UTS), yield strength (YS) and elongation to be determined [11].
Statistical multilinear regression analyses

Two statistical multilinear regression analyses were developed: (1) optical microscope martensite measurements versus steel chemistry and intercritical austenitizing temperature and, (2) tensile properties versus steels chemistry, intercritical austenitizing temperature and austempering temperature. The models were used to determine the role of chemistry and heat treatment conditions on the volume fraction of austenite at high temperature and tensile properties. A statistical analysis was accomplished using MINITAB [12]. Only first and second order interactions with $p$ values $< 0.05$ were taken in account for the multilinear regression models.

Results and Discussion

Determination of the intercritical austenitizing temperatures

As expected, the volume percent of martensite (austenite at high temperature) increased as the intercritical austenitizing temperature increased. The differences in chemical composition produced differences in the amount of martensite obtained at the same intercritical austenitizing temperature (Figure 1). Statistical multilinear regression analyses showed that the amount of martensite at the same intercritical austenitizing temperature increased as carbon content increased and decreased as silicon content increased. This result was expected because a higher carbon concentration decreases the intercritical temperature range and silicon decreases the eutectoid temperature ($A_1$). The quantity of the other alloying elements (nickel, manganese, chromium and molybdenum) did not show a significant statistical influence on the volume percent of martensite at the alloy levels used in this study. Equation 1 was obtained using statistical linear regression analysis of the alloy chemistry, intercritical austenitizing temperature and optical microscopy
martensite measurements. Figure 2 shows the comparison between the experimental values and the values obtained using the linear regression model (Equation 1).

\[
\%\text{Martensite} = -1390.6 + 241.6C - 164.3Si + 1.95IAT + 5.19(C - 0.299)(IAT - 733.2)
\]

where: \(C\) is wt% carbon, \(Si\) is wt% silicon and \(IAT\) is intercritically austenitizing temperature in °C.

The information above was used to define the intercritical austenitizing temperatures to achieve a volume fraction of intercritical austenite between 30% and 40%. The temperatures chosen are listed in Table 3.

The evolution of the microstructure with temperature showed that at temperatures near to \(A_1\) austenite started to nucleate at the grain boundaries and grow into the pearlite grains. These results are in agreement with previous research that has shown that austenite at high temperature nucleates at the ferrite-pearlite grain boundaries and grew into the pearlite grains where more carbon is available [5, 6]. Undissolved pearlite was still present at temperatures near to \(A_1\) after three hours of intercritical austenitizing, which indicated that stage one in the formation of austenite was not yet complete. Increasing the intercritical austenitizing temperature increased the amount of austenite and decreased the amount of undissolved pearlite. At the highest temperatures, dissolution of all the carbides occurred providing a microstructure of ferrite plus austenite. Figure 3 shows representative optical microscope images of the evolution of the microstructure with temperature for the steel 8620. Figure 4 shows secondary electron images SEM images of the same 8620 steel shown in Figure 3. Table 4 lists the phases found in the steels at different intercritical austenitizing temperatures.


**Microstructure**

The microstructures produced by intercritical austempering corresponded to multiphase microstructures with ferritic matrix plus spheroidized carbides, pearlite, bainite and martensite. The pearlite originated from the original microstructure prior to intercritical austenitization while the spheroidized carbides formed during the intercritical austenitizing step of the heat treatment. Upon quenching and holding at the austempering temperature, austenite transformed to bainite. The presence of martensite in the final microstructures indicated that the transformation of austenite to bainite was incomplete, and the remainder of austenite transformed to martensite upon cooling to room temperature. Figure 5 shows representative pictures of the microstructures of the materials in this study. Figure 6 shows secondary electron images of the phases in the materials.

**Tensile properties**

The results indicated that performing intercritical austempered heat treatments to common steel grades produced materials with a relationship between UTS and elongation similar to that obtained with DP and TRIP steels. In some cases, the 8620 steel showed higher elongation than TRIP steels with equivalent UTS. Figure 7 shows the relationship between UTS and elongation for the materials in this study. The results are compared to the values for DP and TRIP steels [4]. The 8620 steel showed the highest elongations (25-34%) and the lowest UTS and YS (525-669 MPa and 229-365 MPa, respectively), followed by the 4330 steel, the cast steel and the 4340 steel, which showed the lowest elongations (17-20%) and the highest UTS and YS (763-963 MPa and 432-489 MPa, respectively). These results suggested that carbon concentration played an important role in the maximum elongation and strengths obtained. Higher carbon concentrations led to
lower elongations and higher strengths and vice versa. Table 5 lists the heat treatment conditions and average tensile properties obtained.

When the same steels used for this study are annealed or quenched and tempered, they exhibit elongation and UTS comparable with the values obtained in this study. However, intercritically austempered heat treatment produced a lower YS/UTS ratio, which suggests that they may have better formability and a higher work hardening rate.

Statistical multilinear regression analyses of steel chemistry, intercritical austenitizing temperature, austempering temperature and tensile properties showed that UTS and YS increased and elongation decreased as the intercritical austenitizing temperature increased, which was explained because higher intercritical austenitizing temperatures produced a higher volume fraction of austenite products in the final microstructures. Austempering temperature did not have an effect on the tensile properties of the 4130 steel and the cast steel. For the 4340 steel, increasing the austempering temperature decreased the UTS and the YS and increased the elongation, while for the 8620 steel the effect was the opposite. These results were explained based on the studies performed by Bakhtiari [8], as follows: (1) for the 4340 steel, increasing the austempering temperature produced a coarser bainite morphology leading to lower strength, (2) for the 8620 steel, incomplete transformation of austenite to bainite and further transformation of austenite to martensite upon cooling to room temperature explained the results. The amount of untransformed austenite increased as the austempering temperature increased, which produced a higher amount of martensite in the materials austempered at higher temperature, and, therefore, higher strength and lower ductility. Equations 2-4 were obtained from the statistical multilinear regression analyses. Figures 8(a)-8(c) show the comparison be-
tween the actual tensile properties and the tensile properties calculated using equations from the statistical analyses.

\[
UTS = -15562 + 6.84IAT + 30.7AT + 18129C + 11633Mn - 10995Si - 48.3AT \times C + 30.8AT \times Si - 32.5AT \times Mn
\]  

Equation 2

\[
YS = 17306 - 23.1IAT + 1.84AT + 3873C - 50931Mn + 65783Si - 10.3AT \times C - 91.2IAT \times Si + 4.48AT \times Si + 68.1IAT \times Mn
\]  

Equation 3

\[
EL = 986 - 1.15IAT - 7.00AT - 711C + 4175Mn - 5067Si + 0.00923IAT \times AT + 1.87AT \times C + 7.31IAT \times Si - 0.699AT \times Si - 5.75IAT \times Mn
\]  

Equation 4

where \(IAT\) is intercritical austenitizing temperature (°C), \(AT\) is austempered temperature (°C). \(UTS\) and \(YS\) are in MPa, elongation in % and concentration of elements \((C, Mn\) and \(Si)\) in weight percent.

**Conclusions**

1. Commercially available alloy steels can be intercritically austempered to produce multiphase microstructures with a ferrite matrix, which showed tensile properties similar to DP and TRIP steels, i.e., low yield strength, high ultimate tensile strength and high elongation.

2. The tensile properties of intercritically austempered steels depended on steel chemistry and austempering temperature. The highest elongations and lowest strengths were achieved with the 8620 steel, while the lowest elongations and highest strengths were obtained with the 4340 steel, which suggested that alloy carbon concentration is related to the maximum strength and elongation achievable.
3. Statistical multilinear regression analyses of tensile properties, steel chemistry, intercritical austenitizing temperature and austempering temperature showed that:

- UTS and YS increased as the intercritical austenitizing temperature increased, which was attributed to an increase in the amount of austenite products.

- The effect of the austempering temperature depended on alloy chemistry. For the 4340 steel, the strength decreased and elongation increased as the austempering temperature increased, which was attributed to bainite coarsening. For the 8620 steel, strength increased and elongation decreased as the austempering temperature increased, which was attributed to incomplete transformation of austenite during the bainitic reaction and further transformation of austenite to martensite upon cooling to ambient temperature.

Acknowledgements

The authors would like to acknowledge the assistance of Rex Heat Treat, Anniston, AL for performing the intercritical austempering at their commercial facility, the expertise of John Griffin (UAB) in tensile test and Dr. Ron O'Malley (Nucor-Decatur) for the chemical analysis. The authors would also like to thank the Falk Corporation, Milwaukee, WI for donating the cast steel used in this study.

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Ricardo Aristizabal is an Assistant Professor of the University of Antioquia in Medellin Colombia; he is currently a PhD candidate at the University of Alabama at Birmingham.
REFERENCES


Table 1. Chemical composition of the steels investigated.

<table>
<thead>
<tr>
<th>Material</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>S</th>
<th>P</th>
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<td>8620</td>
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<td>0.83</td>
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<td>0.74</td>
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<td>1.79</td>
<td>0.83</td>
<td>0.20</td>
<td>0.009</td>
<td>0.007</td>
</tr>
<tr>
<td>Cast steel</td>
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<td>0.88</td>
<td>0.47</td>
<td>0.69</td>
<td>0.80</td>
<td>0.43</td>
<td>0.016</td>
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</table>

Table 2. Austempering temperatures.

<table>
<thead>
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<th>4340</th>
<th>Cast Steel</th>
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<tbody>
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<td>Austempering temperature, °C</td>
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Table 3. Intercritical austenitizing temperatures selected.

<table>
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<td>Intercritical Austenitizing Temperature, °C</td>
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<td>730</td>
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Table 4. Microstructure evolution of the steels under study with temperature after three hours of intercritical austenitizing.

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<th>Temperature, °C</th>
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<td>720</td>
<td>Undissolved pearlite + ferrite + martensite</td>
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<tr>
<td></td>
<td>730</td>
<td>Spheroidized carbide in ferrite + ferrite + martensite</td>
</tr>
<tr>
<td></td>
<td>740</td>
<td>Ferrite + martensite</td>
</tr>
<tr>
<td>4330</td>
<td>710</td>
<td>Undissolved pearlite + martensite</td>
</tr>
<tr>
<td></td>
<td>720</td>
<td>Spheroidized carbide in ferrite + martensite</td>
</tr>
<tr>
<td></td>
<td>730</td>
<td>Ferrite + martensite</td>
</tr>
<tr>
<td>4340</td>
<td>710</td>
<td>Undissolved pearlite + spheroidized carbides in ferrite + martensite</td>
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<tr>
<td></td>
<td>720</td>
<td>Spheroidized carbides in ferrite + martensite</td>
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<tr>
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<td>Undissolved pearlite + spheroidized carbides in ferrite + martensite</td>
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<td></td>
<td>740</td>
<td>Spheroidized carbides in ferrite + martensite</td>
</tr>
<tr>
<td></td>
<td>750</td>
<td>Ferrite + martensite</td>
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Table 5. Heat treatment conditions and average tensile properties obtained.

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<th>Steel</th>
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<th>Austempering Temperature, °C</th>
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<th>YS, MPa</th>
<th>Elongation, %</th>
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Figure 1. Experimental volume percent of martensite (austenite at high temperature) vs intercritical austenitizing temperature for steels intercritically austenitized for 3 hours.

Figure 2. Comparison between the experimental volume percent of martensite (austenite at high temperature) and the volume percent of martensite (austenite at high temperature) obtained with Equation 1.

\[%\text{Martensite} = -1390.6 + 241.6C - 164.3Si + 1.95IAT + 5.19(C - 0.299)(IAT - 7.3)\]

\(R^2 = 0.96\)
Figure 3. Optical microscope images of the evolution of the microstructure of 8620 steel with temperature after three hours of intercritical austenitizing at (a) 720°C, (b) 730°C, (c) 740°C.
Figure 4. Secondary electron images of the evolution of the microstructure of 8620 steel with temperature after three hours of intercritical austenitizing at (a) 720°C, (b) 730°C, (c) 740°C. (F: Ferrite, M: Martensite, UP: Undissolved pearlite, F+C: Ferrite plus spheroidized carbides).
Figure 5. Optical microscope images of intercritically austempered steel. (a) steel 8620 intercritically austenitized at 730°C followed by austempering at 400°C for 10 minutes. (b) steel 4330 intercritically austenitized at 720°C followed by austempering at 400°C for 10 minutes. (c) steel 4340 intercritically austenitized at 715°C followed by austempering at 300°C for 10 minutes. (d) cast steel intercritically austenitized at 730°C followed by austempering at 300°C for 10 minutes.
Figure 6. Secondary electron images of the same intercritically austempered steels shown in Figure 5. (a) steel 8620. (b) steel 4330. (c) steel 4340. (d) cast steel. (F: Ferrite, M: Martensite, B: Bainite, F+C: Ferrite plus spheroidized carbides).
Figure 7. Relationship between ultimate tensile strength and elongation for intercritically austempered steels. The results are compared with the mechanical properties of DP and TRIP steels in reference [4].

Figure 8. Comparison of actual UTS and UTS calculated using Equation 2 obtained with multilinear regression analyses.
Figure 9. Comparison of actual YS and YS calculated using Equation 3 obtained with multilinear regression analyses.

\[ YS = 17306 - 23.14AT + 1.84AT^2 + 3873C - 50931Mn + 65783Si - 10.3AT \times C - 91.2AT \times Si \]
\[ + 4.48AT \times Si + 68.11AT \times Mn \]

\[ R^2 = 0.9863 \]

Figure 10. Comparison of actual elongation and elongation calculated using Equation 4 obtained with multilinear regression analyses.

\[ EL = 98.6 - 1.151AT - 7.004AT - 7711C + 4175Mn - 50675Si + 0.049231AT \times AT + 1.87AT \times C \]
\[ + 7.31AT \times Si - 0.699AT \times Si - 5.751AT \times Mn \]

\[ R^2 = 0.9502 \]
6. INTERCRITICALLY AUSTENITIZED QUENCHED AND TEMPERED DUCTILE IRON

by

RICARDO E. ARISTIZABAL, ROBIN D. FOLEY AND ALAN P. DRUSCHITZ

In preparation for International Journal of Metalcasting

Format adapted for dissertation
Abstract

Ductile iron with a matrix of ferrite plus martensite was produced using a ductile iron alloyed with ~0.7 wt% manganese and ~0.5 wt% nickel. Three different volume percents of martensite (16, 24 and 37 vol%) were formed by austenitizing in the intercritical region (ferrite+austenite) followed by quenching in a polymeric solution to room temperature. The materials were evaluated in the untempered condition and after tempering at 400°C and 500°C for one hour. This paper reports the effects of volume percent of martensite and tempering conditions on microstructure and tensile properties. Semi-quantitative chemistry and microhardness of ferrite and martensite were also determined as a function martensite volume percent. The results indicated that the carbon available to form the austenite during intercritical austenitizing came mainly from the carbon in the matrix and that partitioning of substitutional elements during intercritical austenitizing is limited. The results also showed that ultimate strength, yield strength and hardness increased and elongation decreased as the volume fraction of martensite increased. Tempering significantly increased the elongation with a only a small decrease in the strengths.

Key words: Intercritical austenitizing, tempering, ductile iron, dual phase.
**Introduction**

Heat treatments can be performed on ductile iron to increase strength, wear resistance, ductility, toughness, and/or improve machinability by controlling the matrix microstructure. For example, annealing produces a fully ferritic matrix, which is necessary when high ductility is required. Normalizing is performed in order to obtain a fully pearlitic matrix for the high strength ductile iron grades. Quenching and tempering provides a fully tempered martensite matrix which provides high strength and excellent wear resistance. Austempering produces an ausferrite matrix which offers exceptional combinations of strength, toughness and wear resistant properties [1-2].

Intercritical heat treatments can also be performed on ductile iron [3-9]. In ferrous alloys, an intercritical heat treatment starts with partial austenitization in the intercritical region where ferrite and austenite are present. The amount of austenite depends on the chemistry of the alloy and the temperature [3-4]. After intercritical austenitizing, the material can be quenched in a salt bath to a temperature above the martensite starting temperature (austempering, typically between 230°C and 400°C) and held for a certain period of time to stabilize the high temperature austenite upon quenching to room temperature. This heat treatment produces a microstructure of graphite nodules in a matrix of ferrite plus austenite particles, which provides a desirable combination of high yield strength, high ultimate tensile strength and high elongation to fracture. This material is known as intercritically austempered ductile iron (IADI) [3-6] or dual phase austempered ductile iron [7-15]. Ductile iron can also be quenched to a temperature below the martensite finish temperature after intercritical austenitizing [16-18]. In this case, the high temperature austenite transforms to martensite. After quenching, the material is tempered in
order to soften and toughen the hard and brittle as-quenched martensite. The resulting microstructure consists of graphite nodules in a matrix of ferrite plus tempered martensite. In this study, such material is called intercritical austenitized quenched and tempered ductile iron (IAQ&TDI).

Previous research has been done in order to produce ferrite-martensite microstructures in unalloyed ductile iron. Rashidi et al. (2000) investigated the effect of tempering conditions in ductile iron having a ferritic-martensitic microstructure. The material was austenitized at 900°C for 480 seconds and then quenched in water. The results indicated that impact strength and elongation increased when tempering was performed at 400°C-500°C for 60-120 minutes, whereas ultimate tensile strength and yield strength remained almost unchanged [16].

Kocatepe et al. (2006) studied the effect of martensite volume fraction and its morphology on the tensile properties of ductile iron with ferritic-martensitic matrix. The material was austenitized between 780°C and 840°C for 30 seconds and then quenched in oil at 100°C. After that, the samples were tempered at 550°C for 1-3 hours. The results indicated that ferritic-martensitic microstructures in ductile iron provided larger elongation than fully martensitic microstructures. Also, strength increased and elongation decreased as the volume fraction of martensite increased and strength increased as martensite particle size decreased [17].

Cerah et al. (2005) produced ferrite-martensite microstructures in ductile iron by intercritically austenitizing at 795°C-815°C for 20 minutes followed by quenching in oil at 100°C. After that, the material was tempered at 500°C for 1h-5h. The results indicated that the variation of strength and ductility with tempering time are related to the inter-
critical austenitizing temperature, which controls the carbon content of the martensite [18].

In this study, IAQ&TDI was produced using a ductile iron alloyed with manganese and nickel (0.7 wt% and 0.5 wt%, respectively). The appropriate intercritical austenitizing temperatures to produce a matrix of continuous ferrite plus martensite were determined experimentally. The effect of the volume fraction of martensite on the tensile properties was investigated. The material was evaluated at three different tempering conditions: untempered, tempered at 400°C for one hour, and tempered at 500°C for one hour. The semi-quantitative composition and microhardness of ferrite and martensite were determined as a function of martensite volume percent.

**Materials and Methods**

*Materials*

The ductile iron used for this study was cast at the University of Alabama at Birmingham in an induction furnace. A seventy pound heat was produced using low carbon steel punchings, granular SiC, granular carbon raiser, ferromanganese, copper turnings and nickel shot. Ductile iron treatment was performed in an open ladle using the sandwich method. Approximately 1.85% magnesium-ferrosilicon (6% Mg) was first added to a pre-heated treatment ladle followed by approximately 0.85% ferrosilicon alloys and approximately 3% cover steel. The ferrosilicon alloys were a combination of foundry grade 75% ferrosilicon alloy (nominally 75% Si, 0.8% Ca, 1% Al) and a barium containing 75% ferrosilicon inoculant that also contained Ca and Al. The base iron was tapped at ~1520°C onto the treatment alloys. The treated iron was slagged-off, a chemistry sample taken and Y-blocks poured at ~1450°C. The Y-block molds were made from chemically
bonded sand using 1.5% binder. Figure 1 shows a drawing with the dimensions of the Y-Blocks produced. The castings were shaken out ~1 hour after pouring. The chemistry of the alloy was determined by optical emission spectroscopy (OES) of a chilled sample. Carbon and sulfur content were determined by combustion analysis using a Leco C/S analyzer. Table 1 lists the chemical composition of the ductile iron used for this study.

**Determination of the intercritical austenitizing temperatures**

Intercritical austenitizing temperatures were determined by placing 13 mm thick samples of the alloy in a pre-heated laboratory furnace (745°C-770°C), held 2.5 hours at temperature, and quenched in water. The samples were tempered at 200°C for one hour to facilitate metallographic examination. Samples were sectioned, polished using standard metallographic techniques and the volume percent of martensite determined by image analysis. Twenty randomly selected fields of view were examined at a magnification of 500X. The information was used to determine the appropriate intercritical austenitizing temperature to obtain a matrix of interconnected ferrite plus martensite.

**Heat treatment**

Rectangular bars 13 x 25 mm, 165 mm long were intercritically austenitized at 750, 760 and 770°C for 2.5 hours and then quenched in a polymeric solution UCON E 20% at ambient temperature. Three different tempering conditions were evaluated: untempered, tempered at 400°C for one hour, and tempered at 500°C for one hour.

Microstructure was characterized using optical microscopy and standard metallographic techniques with a 1μm diamond final polish and a 3% nital etch. Semi-quantitative compositions of martensite and ferrite were obtained using electron disper-
sive spectroscopy (EDS) with a scanning electron microscope (SEM) at a 30 kV accelerating voltage. The analyses were performed on the untempered samples. Sixteen analyses were performed in each phase per sample. Martensite and ferrite microhardness was determined using a Buehler Micromet II Microhardness tester. Ten Vickers hardness indentations were done in each phase per sample with a load of 25 grams. X-Ray diffraction (XRD) was performed using a Siemens D-500 Diffractometer with Cu radiation. The scans were performed for 2θ between 40° and 90° with a step size of 0.02° and a dwell time of 5 seconds. The results were used to determine if some retained austenite existed in the untempered samples.

Tensile bars, 9 mm diameter x 36 mm gage length were machined from the heat treated rectangular bars and tested in accordance to ASTM E8 on an MTS Model 810 servo hydraulic tension/compression tester [19]. Brinell hardness was determined using a NewAge model HB3000B hardness tester with Lab B.O.S.S model #OS100 optical reader and C.A.M.S software. A 10 mm diameter steel ball and a load of 3,000 kg were used for the hardness measurements.

Results and Discussion

Determination of the intercritical austenitizing temperatures

As expected, the volume percent of martensite increased as the intercritical austenitizing temperature increased. Martensite volume percent varied from 12 vol% at 745°C to 37 vol% at 770°C. The comparison of the intercritical austenitizing temperatures with previously published data [16-18] showed that the alloy in this study can be intercritical austenitized at lower temperatures than unalloyed ductile iron to obtain similar volume percentages of martensite. This result was explained by the presence of man-
ganese and nickel, which lower the eutectoid temperature \( (A_1) \). Figure 2 shows the variation of the martensite volume percent with intercritical austenitizing temperature.

**Microstructure**

Intercritical austenitizing followed by quenching and tempering produced microstructures consisting of graphite nodules in a matrix of ferrite plus tempered martensite (Figure 3). The results indicated that the austenite at high temperature started to nucleate at the eutectic cells, which occurred because during the solidification process the manganese (which is an austenite stabilizing element) segregates to the eutectic cells [3], and favors the nucleation of the austenite in these regions. The areas surrounding the graphite nodules were mostly ferritic. This result was attributed to segregation of silicon (which is a ferrite stabilizer) during the solidification process to the areas surrounding the graphite nodules [3]. XRD results showed that no retained austenite was present in the untempered condition (Figure 4).

**Semiquantitative chemistry of martensite and ferrite**

The results of the semiquantitative chemistry analyses of the phases are shown in Figure 5 and listed in Table 2. Error bars correspond to the 95% confidence interval. Statistically, the concentration of silicon, manganese, nickel and copper in martensite and ferrite did not change as a function of the martensite volume percent (or intercritical austenitizing temperature). Similar results have been found in dual phase steels [20]. The results indicated limited partitioning of substitutional elements during intercritical austenitizing, which is explained by the low rate of substitutional diffusion at the temperatures of the heat treatment. Silicon, nickel and copper concentrations were higher and manga-
nese concentration was lower in the ferrite than in the martensite. As was mentioned earlier, austenite started to nucleate at the eutectic cells, and ferrite was found in the areas surrounding the graphite nodules. It has been well established that manganese segregates during solidification to the eutectic cells, while silicon, nickel and copper segregate to the areas surrounding the graphite nodules [3]. Therefore, the difference in chemistry between the martensite and the ferrite phases in the material in this study was due to segregation during the solidification process of the material rather than diffusion during the heat treatment.

**Microhardness**

The microhardness of the martensite was a function of both the intercritical austenitizing temperature and the tempering condition (Figure 6). The microhardness of the martensite decreased as the volume percent of martensite in the material increased and as the tempering temperature increased. The XRD results showed no retained austenite was detected in the untempered samples. Therefore, the higher microhardness of the martensite in the materials with lower martensite volume percent indicated that, as the martensite volume percent in the material increased, the concentration of carbon in the martensite decreased. This result showed that the carbon available to form austenite during intercritical austenitizing came mainly from the carbon present in the pearlite prior to the heat treatment and that the diffusion of carbon from the graphite nodules is limited during intercritical austenitizing, as suggested by Druschitz, et al. [4]. Previous published data indicated that carbon content in the martensite increased as intercritically austenitized temperature increased [18]. However, in that study, the matrix before heat treatment was fully ferritic. As a result, the only carbon available to form the austenite came from the
graphite nodules. In the present work, the as-cast microstructure contained pearlite, which provided the carbon for the austenite formation.

The microhardness of the ferrite was a function of the volume percent martensite (Figure 7). As the martensite volume percent increased, ferrite microhardness decreased. As stated previously, the manganese, silicon, nickel and copper concentrations in the ferrite were the same regardless of the volume percent martensite in the material (or intercritical austenitizing temperature). Also, it is a well-known fact that carbon solubility in ferrite is low. Therefore, no differences in ferrite carbon content were expected between materials with different amounts of martensite. As a consequence, the differences in ferrite microhardness cannot be explained by differences in chemical composition (solid solution hardening). Consequently, the results of the ferrite microhardness may be interpreted as an effect of residual stresses in the ferrite matrix caused by the formation of the martensite. As was mentioned previously, the results of the microhardness of the martensite indicated that the lower the martensite volume percent, the higher its carbon content. The austenite to martensite transformation produces a volume expansion [21]. Higher carbon contents in the austenite produce a larger volume change during the austenite to martensite transformation, which leads to a higher residual stress in the surrounding ferrite. This may explain the higher microhardness of the ferrite in the materials with lower volume percent martensite. After tempering, the difference in the microhardness of the ferrite between samples with different amounts of martensite drops significantly due to relief of the residual stress, which supports the hypothesis of the presence of residual stresses in the ferrite in the untempered samples.
Tensile properties and hardness

Elongation decreased and hardness, ultimate tensile strength (UTS) and yield strength (YS) increased as the volume percent martensite increased for all the tempering conditions evaluated (Figures 8-11 and Table 3). The materials in the untempered condition had the lowest elongation (4.9%-7.4%) due to the presence of brittle martensite in the microstructure. Tempering at 400°C for one hour increased the elongation between 1.7 and 1.9 times with no effect on the ultimate tensile strength and the yield strength. Tempering at 500°C for one hour increased the elongation between 2 and 2.5 times, slightly decreased the ultimate tensile strength and had no effect on the yield strength. These results agree with previous studies which indicate that tempering at 400°C-500°C increases elongation without affecting strength and that strength increases as volume fraction of martensite increases [16-17].

IAQ&TDI with 16 vol% martensite had an elongation 3.7 times larger than the minimum elongation of ductile iron with a pearlitic-ferritic matrix (Grade D500), yet similar ultimate tensile strength and yield strength. IAQ&TDI with 24 vol% and 37 vol% of martensite had an elongation 2.5-2.7 times larger than the minimum elongation of ductile iron with a pearlitic-ferritic matrix (Grade D550) yet higher ultimate tensile strength (up to 89 MPa) and higher yield strength. Compared to ductile iron with a ferrite-pearlite matrix, IAQ&TDI had higher ultimate tensile strength (up to 140 MPa), higher yield strength (up to 47 MPa), yet similar elongation. These results can be explained by (1) the ferrite plus tempered martensite matrix of IAQ&TDI which provides a good combination of elongation and strength and (2) the solid solution strengthening produced by manganese and nickel as alloying elements. Figure 12 shows the relationship between elonga-
tion and ultimate tensile strength for ductile iron with different microstructures [22], including the data obtained for IAQ&TDI in this study.

Conclusions

1. The concentrations of silicon, manganese, nickel and copper in martensite and ferrite were determined by the segregation of these elements during the solidification process of the alloy rather than by diffusion during the intercritical austenitizing step of the heat treatment.

2. Martensite microhardness was a function of intercritical austenitizing temperature. As the intercritical austenitizing temperature increased the martensite volume percent increased and the martensite microhardness decreased, which suggested that the concentration of carbon in the martensite decreased. This result indicates that matrix carbon rather than nodules was the primary carbon source in martensite.

3. Ferrite microhardness indicated the presence of residual stresses in the ferrite phase in the untempered condition caused by the volume expansion of the austenite to martensite transformation. Tempering relieved the residual stress in the ferrite.

4. The tensile properties and hardness of IAQ&TDI ductile iron were a function of the volume fraction of martensite and tempering conditions. As the volume fraction of martensite increased, ultimate tensile strength, yield strength and hardness increased and elongation decreased. Tempering increased the elongation and decreased hardness with only a small effect on the ultimate tensile strength and with no effect on the yield strength.
5. IAQ&TDI had ultimate tensile strength and yield strength similar to pearlitic ductile irons but had higher elongation (2.5 to 3.7 times higher). These properties are provided by first, a microstructure of well-formed graphite nodules in a matrix of ferrite plus particles of tempered martensite and second, the solid solution strengthening of manganese and nickel used as alloying elements.

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Ricardo Aristizabal is an Assistant Professor of the University of Antioquia in Medellin Colombia; he is currently a PhD candidate at the University of Alabama at Birmingham.
REFERENCES


### Table 1. Chemical composition of the iron investigated.

<table>
<thead>
<tr>
<th>Element, wt%</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Ni</th>
<th>Cu</th>
<th>Mg</th>
<th>S</th>
<th>P</th>
<th>Cr</th>
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<td></td>
<td>3.87</td>
<td>2.33</td>
<td>0.69</td>
<td>0.50</td>
<td>0.19</td>
<td>0.05</td>
<td>0.016</td>
<td>0.019</td>
<td>0.04</td>
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### Table 2. Results of the semiquantitative chemistry analyses in wt% of ferrite and martensite.

<table>
<thead>
<tr>
<th>Phase</th>
<th>Element</th>
<th>Ferrite</th>
<th>Martensite, vol%</th>
<th>Martensite</th>
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<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>16</td>
<td>24</td>
</tr>
<tr>
<td>Ferrite</td>
<td>Si</td>
<td>3.30 ± 0.04</td>
<td>3.38 ± 0.07</td>
<td>3.42 ± 0.06</td>
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<tr>
<td></td>
<td>Mn</td>
<td>0.76 ± 0.04</td>
<td>0.74 ± 0.04</td>
<td>0.73 ± 0.05</td>
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<tr>
<td></td>
<td>Ni</td>
<td>0.71 ± 0.03</td>
<td>0.73 ± 0.03</td>
<td>0.76 ± 0.03</td>
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<tr>
<td></td>
<td>Cu</td>
<td>0.22 ± 0.02</td>
<td>0.23 ± 0.02</td>
<td>0.25 ± 0.02</td>
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<tr>
<td>Martensite</td>
<td>Si</td>
<td>1.99 ± 0.1</td>
<td>2.10 ± 0.11</td>
<td>2.18 ± 0.11</td>
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<td></td>
<td>Mn</td>
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<td>1.78 ± 0.17</td>
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<tr>
<td></td>
<td>Ni</td>
<td>0.53 ± 0.03</td>
<td>0.49 ± 0.03</td>
<td>0.52 ± 0.03</td>
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<tr>
<td></td>
<td>Cu</td>
<td>0.13 ± 0.02</td>
<td>0.10 ± 0.02</td>
<td>0.11 ± 0.01</td>
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</table>

### Table 3. Mechanical properties of IAQ&TDI

<table>
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<tr>
<th>Austenitizing Temperature, °C</th>
<th>Martensite, vol%</th>
<th>Tempering Condition</th>
<th>UTS, MPa</th>
<th>YS, MPa</th>
<th>Elongation, %</th>
<th>HBN</th>
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<tbody>
<tr>
<td>750</td>
<td>16</td>
<td>Untempered</td>
<td>532</td>
<td>329</td>
<td>7.4</td>
<td>197</td>
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<tr>
<td></td>
<td></td>
<td>400°C for 1 hour</td>
<td>524</td>
<td>325</td>
<td>14.3</td>
<td>187</td>
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<tr>
<td></td>
<td></td>
<td>500°C for 1 hour</td>
<td>513</td>
<td>327</td>
<td>15.5</td>
<td>175</td>
</tr>
<tr>
<td>760</td>
<td>24</td>
<td>Untempered</td>
<td>605</td>
<td>353</td>
<td>5.8</td>
<td>216</td>
</tr>
<tr>
<td></td>
<td></td>
<td>400°C for 1 hour</td>
<td>609</td>
<td>363</td>
<td>10.3</td>
<td>209</td>
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<tr>
<td></td>
<td></td>
<td>500°C for 1 hour</td>
<td>585</td>
<td>371</td>
<td>11.8</td>
<td>195</td>
</tr>
<tr>
<td>770</td>
<td>37</td>
<td>Untempered</td>
<td>643</td>
<td>376</td>
<td>4.9</td>
<td>241</td>
</tr>
<tr>
<td></td>
<td></td>
<td>400°C for 1 hour</td>
<td>677</td>
<td>406</td>
<td>8.5</td>
<td>226</td>
</tr>
<tr>
<td></td>
<td></td>
<td>500°C for 1 hour</td>
<td>600</td>
<td>377</td>
<td>12.0</td>
<td>204</td>
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Figure 1. Dimensions of the Y-Block used to produce the ductile iron castings.

Figure 2. Volume percent of martensite as a function of intercritical austenitizing temperature for IAQ&TDI.

Figure 3. Representative microstructures of IAQ&TDI intercritically austenitized for 2.5 hours at (a) 750°C (16 vol.% martensite) (b) 760°C (24 vol.% martensite), and (c) 770°C (37 vol.% martensite).

Figure 4. XRD spectrums of untempered IAQ&TDI with different amounts of martensite.
Figure 5. Semiquantitative chemistry of ferrite and martensite phases as function of volume percent of martensite.

Figure 6. Effect of volume percent of martensite and tempering conditions on the microhardness of the martensite phase.

Figure 7. Effect of volume percent of martensite and tempering conditions on the micro-hardness of the ferrite phase.

Figure 8. Effect of volume percent of martensite and tempering conditions on elongation of IAQ&TDI.

Figure 9. Effect of volume percent of martensite and tempering conditions on ultimate tensile strength of IAQ&TDI.


Figure 10. Effect of volume percent of martensite and tempering conditions on yield strength of IAQ&TDI.

Figure 11. Effect of volume percent of martensite and tempering conditions on hardness of IAQ&TDI.


Figure 12. Relationship between elongation and ultimate tensile strength for ductile iron with different microstructures.

7. INTERCRITICALLY AUSTEMPERED DUCTILE IRON

by

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Abstract

In this study four ductile irons alloyed with nickel and manganese were prepared and intercritically austempered to obtain materials with different volume fractions of austenite. The influence of alloy chemistry and intercritical austenitizing temperature on the stability of the austenite during deformation was studied using neutron diffraction and x-ray Diffraction (XRD). Lattice strains of the austenite and ferrite phases and microstructure, hardness and tensile properties of the materials were determined as well. The results showed that austenite morphology, ferrite lattice strain and austenite stability depended upon alloy chemistry. Tensile properties were a function of alloy chemistry and intercritical austenitizing temperature.

Key words: Intercritical austenitizing, austempering, neutron diffraction, austenite stability.
Introduction

Intercritical heat treatment in ductile iron is a two step heat treatment that starts with austenitizing in the intercritical temperature range, where ferrite and austenite coexist. After intercritical austenitizing, the material can be quenched to a temperature below the martensite starting temperature and then tempered, which produces a microstructure of graphite nodules in a matrix of ferrite plus tempered martensite [1-4]. The material can be also fast cooled from the intercritical austenitizing temperature to a temperature above the martensite starting temperature (austempered), typically between 230°C-400°C. This heat treatment provides a microstructure of graphite nodules in a matrix of ferrite plus austenite particles. The austenite particles can be present as untransformed austenite volumes or as austenite particles in ausferrite. This material is known as intercritically austempered ductile iron (IADI) [5-7] or dual phase austempered ductile iron [8-16].

IADI is a viable alternative to produce powertrain and chassis components for the automotive and military industry, since it possesses high yield strength (~500 MPa), high ultimate tensile strength (~750 MPa), high elongation to fracture (~14%-20), good fracture toughness (~45-55 MPa-m$^{1/2}$), good resistance to environmental cracking, excellent castability and excellent machinability [16-28].

The mechanical properties of IADI are related to the volume fraction of austenite in the matrix, which is determined for a given chemistry by the intercritical austenitizing temperature chosen. Increasing the intercritical austenitizing temperature increases the amount of austenite (ausferrite) in the matrix, leading to higher ultimate tensile strength, yield strength, and fracture toughness but lower elongation [8-10].
The stability of the austenite during deformation plays an important role in the mechanical properties of ferrous materials because it can increase the elongation and the work hardening coefficient by an effect known as transformation induced plasticity, which is caused by the transformation of the austenite to martensite due to a deformation process. The transformation of the austenite has been found to be beneficial to the elongation when it takes place at relatively high strains, while austenite with low stability that transform to martensite at low strains does not contribute to the elongation [29]. Also, it has been found that chemistry, morphology and size of the austenite are the main factors that determine its stability during deformation. Low carbon content and large austenite particles tend to transform at very low strains and do not increase the elongation, while high carbon content and small austenite particle favor the stability of the austenite during deformation. Also, retained austenite between the subunits of bainite is more stable than blocky austenite [29-30]. In regular austempered ductile iron (ADI), the formation of strain induced martensite has been found to depend upon the austenitizing and austempering temperatures. High austenitizing and austempering temperatures favor the formation of low carbon blocky austenite, which transforms to martensite during deformation at small strains. On the other hand, low austenitizing and austempering temperatures produce small austenite particles with high carbon concentration that are more stable [31].

The stability and elastoplastic properties of austenite in multiphase and austenitic stainless steels have been studied using a variety of techniques, which include neutron diffraction [32-37]. The elastoplastic properties of the phases in multiphase steels are related to the thermomechanical processing conditions and the rate of transformation of the austenite to martensite during deformation does not depend upon the amount of initial
retained austenite on the material [32-34]. Jacques et al. studied multiphase steels during tensile loading and found that the yield strength of the austenite increases as the carbon content increases [35]. Oliver et al. studied the stress induced martensitic transformation in TRIP steels and found that the austenite grains having the ⟨100⟩ parallel to axial load transforms preferentially. They also found that the load is transferred from the original austenite to the evolving martensite during plastic deformation [36].

In IADI the carbon content of the high temperature austenite is determined by the intercritical austenitizing temperature. Studies performed in unalloyed ductile iron with ferritic matrices prior to heat treatment have shown that the carbon to form the austenite at high temperature comes from the graphite nodules; and, therefore, the carbon content in the austenite increases as the intercritical austenitizing temperature increases [10-11]. Some authors have used ductile iron with ferritic-pearlitic matrices as starting microstructures [4-7, 17-25]. Druschitz et al. suggested that under this condition, the carbon to form the austenite at high temperature comes from the carbon in the pearlite and the diffusion of carbon from the graphite nodules is limited. Consequently, the concentration of carbon in the austenite at high temperature decreases as the intercritical austenitizing temperature increases [5]. As it happens in steels, the segregation of other alloying elements such as silicon, nickel and manganese during intercritical austenitizing may be limited due to the low rate of substitutional diffusion at the temperatures of the heat treatment [38]. As a result, the concentration of these elements in the austenite is determined mainly by the chemistry of the alloy and their segregation during the solidification process.

Therefore, it is expected that variations in alloy chemistry and intercritical austenitizing
temperature have an effect on the austenite chemistry and therefore an influence on its stability during deformation.

In this study four ductile irons alloyed with nickel and manganese were prepared and intercritically austempered to obtain materials with different volume fractions of austenite. The influence of alloy chemistry and intercritical austenitizing temperature on the stability of the austenite during deformation was studied using neutron diffraction and x-ray diffraction (XRD). Lattice strains of the austenite and ferrite phases, microstructure, hardness and tensile properties of the obtained materials were determined as well.

Materials and Methods

Materials

Four different ductile irons alloyed with nickel and manganese were cast at the University of Alabama at Birmingham in an induction furnace. Seventy-pound heats of each alloy were produced. Low carbon steel punchings, granular SiC, granular carbon raiser, ferromanganese, copper turnings and nickel shot were used to prepare the alloys. Ductile iron treatment was performed in an open ladle using the sandwich method. Approximately 1.5% magnesium-ferrosilicon (6% Mg) was first added to a pre-heated treatment ladle followed by approximately 1.0% ferrosilicon alloys and approximately 3% cover steel. The ferrosilicon alloys were a combination of foundry grade 75% ferro-silicon alloy (nominally 75% Si, 0.8% Ca, 1% Al) and a barium containing 75% ferrosilicon inoculant that also contained Ca and Al. The base iron was tapped at ~1480°C-1540°C onto the treatment alloys. The treated iron was slagged-off, a chemistry sample taken and Y-blocks poured at ~1400°C-1450°C. The Y-block molds were made from chemically bonded sand using 1.0% binder (Figure 1). The castings were shaken out ~1
hour after pouring. The chemistry of the alloys was determined by optical emission spectroscopy (OES) of a chilled sample. Carbon and sulfur concentrations were determined by combustion analysis using a Leco C/S analyzer. Table 1 lists the chemical composition of the ductile iron alloys used for this study.

*Heat treatment*

Rectangular bars 12.7 x 25 mm, 165 mm long were intercritically austenitized at 720°C-770°C for 4 hours in a nitrogen atmosphere and then austempered in a neutral salt bath at 357°C for 2.25 hours. Two different intercritical austenitizing temperatures were used for each alloy in order to obtain different amounts of austenite. Microstructure was determined using optical microscopy and standard metallographic techniques with a 1μm diamond final polish. The samples were etched with 3% Nital (3% nitric acid in methanol). Brinell hardness was determined using a NewAge model HB3000B hardness tester with Lab B.O.S.S model #OS100 optical reader and C.A.M.S software. A 10 mm diameter steel ball and a load of 3,000 kg were used for the hardness measurements.

Tensile bars, 9 mm diameter x 36 mm gage length were machined from the heat treated rectangular bars and tested in accordance to ASTM E8 on an MTS Model 810 servo hydraulic tension/compression tester [39]. After tensile testing, samples for XRD were taken from the grip and from the gage (close to the fracture surface) of the regular tensile specimens. The samples taken from the grip were considered unloaded/unstrained and the samples taken from the gage were considered loaded/strained. XRD were performed using a Siemens D-500 Diffractometer with Cu radiation. The scans were performed for 2θ between 20° and 90° with a step size of 0.04° and a dwell time of 30 seconds. XRD results were used to determine austenite volume percent before and after
loaded/strained and the austenite lattice parameter. Quantitative analyses were conducted using JADE wherein the two theta zero, background, scale, lattice parameter, preferred orientation, peak shape, U (peak sharpness) and W (peak width) were refined [40]. The space group, atomic positions and starting cells for BCC and FCC iron phases and graphite were obtained from the PDF database (04-002-1253, 00-052-0512 and 00-056-0160, respectively) [41]. Carbon content in the austenite of the unloaded/unstrained samples was estimated from the lattice parameter using Equation 1 which provides an approximate value of the carbon content in the austenite that was used for comparison between samples [42].

\[ \%C = \left( a_\gamma - 3.555 \right) / 0.044 \]  

Equation 1

Special tensile specimens, 4 mm diameter x 25 mm gage length, for neutron diffraction studies were machined. The gage length of the tensile specimens for neutron diffraction were polished with progressively finer grit papers (240, 320, 400, 600 grit), then 1 micron diamond paste and finally 0.05 micron alumina to remove any deformation or transformation that may have occurred on the machined surfaces. Neutron diffraction studies were performed using a load frame at the NRSF2 at HFIR at Oak Ridge National Laboratory (ORNL). Axial load was incrementally increased, and diffraction data was obtained parallel and perpendicular to the loading axis at each load level. The information was collected for the ferrite BCC110 and BCC200 reflections and for the austenite FCC111 and FCC311 reflections. This allowed the in-situ determination of the lattice strain of the individual phases (ferrite and austenite). The plane specific diffraction elastic
constants ($\text{DEC}_{hkl}$) were obtained from the slope of the elastic region of the lattice strain versus applied stress data. Lattice strains were calculated using Equation 2.

\[
\text{Lattice strain, ppm} = \frac{(d_{hkl} - d_{hkl}^0)}{d_{hkl}^0} \times 10^6 \quad \text{Equation 2}
\]

where $d_{hkl}$ is the lattice spacing of a particular plane when loaded and $d_{hkl}^0$ is the lattice spacing of that plane at no load.

Determination of the stability of the austenite phase under load conditions was also performed. A measure of the amount of austenite at each load was calculated from the integrated intensities areas of the reflections FCC111, FCC311, BCC110 and BCC200 using Equation 3. The amount of austenite remaining ($\Delta$) at each applied load compared to that present at no load was determined using Equation 4.

\[
\gamma = \frac{(\text{FCC}111 + \text{FCC}311)}{(\text{FCC}111 + \text{FCC}311) + (\text{BCC}110 + \text{BCC}200)} \times 100 \quad \text{Equation 3}
\]

\[
\Delta = \frac{\gamma_{\text{load}}}{\gamma_{\text{no load}}} \times 100 \quad \text{Equation 4}
\]

Results and Discussion

Microstructure

The microstructures consisted of graphite nodules in a matrix of ferrite plus particles of austenite and/or ausferrite. Spheroidized carbides were observed in the microstructure of the samples intercritically austenitized at lower temperatures of each alloy, which corresponded to undissolved pearlite from the as-cast microstructure. The high manganese IADI showed untrasformed austenite volumes as blocky particles located along the intercellular boundaries, which was consistent with previous published results.
obtained with regular ADI. These studies demonstrated that the combination of the strong segregation of manganese and its effect delaying the bainitic reaction lead to the formation of blocky low carbon austenite in the intercellular boundaries [43-44]. The austenite in the nickel and the manganese-nickel IADI alloys was a combination of plate-like austenite between bainitic ferrite particles (ausferrite) and some untransformed austenite volumes, which may be a consequence of the austenite stabilizing effect of nickel. Figure 2 shows representative optical microscope pictures of the heat-treated microstructures. Figure 3 shows representative SEM secondary electron images of the austenite particles found in the materials under study.

X-ray diffraction

The results of the XRD measurements showed that, as expected, the amount of austenite increased as the intercritical austenitizing temperature increased for all the alloys under study. The estimated carbon content of the austenite in the unload-ed/unstrained samples indicated that the austenite in the low nickel alloy IADI had the highest carbon concentration and the austenite in the high manganese IADI had the lowest. The results indicated no austenite transformed to martensite during loading in the low nickel alloy intercritically austenitized at 750°C and the high nickel alloy intercritically austenitized at 720°C (lower intercritically austenitizing temperatures for these alloys). For all the other alloy-heat treatment combinations the amount of austenite decreased after loading, which indicated that some of the austenite transformed to martensite during deformation. XRD results are listed in Table 2. The reported values are the average of the measurements performed on two samples, one parallel and one perpendicular to the axial load.
Tensile properties and hardness

As expected, ultimate tensile strength, yield strength and hardness increased and elongation decreased as the volume percent of austenite (ausferrite) increased for all the alloys under study, which agrees with previous published results [8, 10]. The high manganese IADI had low elongation (4.0-6.5%), which was attributed to the segregation of manganese to the intercellular boundaries. Manganese delays the formation of ausferrite, so that large untransformed austenite volumes were present at the intercellular boundaries. This has been found to decrease the ductility of regular ADI [43, 44]. Nickel and manganese-nickel alloyed ductile irons showed high elongation (14.7%-19.2%). In conventional ADI, manganese is usually limited to 0.4 wt% because of its detrimental effect on the ductility [45]. The results indicated that IADI can withstand higher manganese concentrations than conventional ADI without decreasing the elongation. It was also found that in the low nickel alloyed ductile iron, increasing the volume percent of austenite significantly increased the ultimate tensile strength (up to 261 MPa) and the yield strength (up to 151 MPa) with only a small effect on the elongation (around 2%). The low nickel alloy intercritically austenitized at 770°C (21wt% austenite) had an ultimate tensile strength of 777 MPa with 16% elongation and 498 MPa yield strength, which was considered the best combination of tensile properties obtained on this study. Table 3 lists the tensile properties and hardness obtained. Figures 4-5 show the results of the tensile test.

Nickel and manganese-nickel alloyed IADI had ultimate tensile strength and yield strength higher than the minimum requirements for pearlitic ductile iron (Grade D700) yet higher elongation. Compared with regular ADI, IADI showed lower ultimate tensile
strength and yield strength and much higher elongation. Figure 6 shows the relationship between ultimate tensile strength and elongation for ductile iron with different microstructures [46], including the data obtained for IADI in this study.

*Neutron diffraction*

As expected, lattice strain increased as stress increased for the planes parallel to the axial load. Lattice strains showed the two stages of the deformation process. The first stage corresponded to pure elastic deformation, where lattice strain increased linearly as stress increased. The slope of the curve of lattice strain vs stress in the elastic region corresponded to the specific diffraction elastic constants for the specific $hkl$ reflections ($DEC_{hkl}$). The second deformation stage started when increasing the stress produced a non-linear increase in the lattice strain, which corresponded to the plastic deformation regime [33]. It was observed for all the alloys and heat treatment conditions that the plastic deformation at the lattice level for the reflections under study started before the macroscopic yield strength of the alloys was reached. Similar results have been obtained using neutron diffraction to study the lattice strain evolution of austenite in stainless steel under tensile loading [37]. Figure 7 shows the lattice strains evolution with applied stress for the low nickel IADI alloy intercritically austenitized at 750°C, which is representative of all the alloys and heat treatment conditions under study in the direction parallel to the load axis.

Agreement between actual $DEC_{hkl}$ and $DEC_{hkl}$ based on single crystal elastic constants calculations were good for the ferrite phase and poor for the austenite phase. The poor agreement for the austenite $DEC_{hkl}$ may be due to shielding of the austenite phase by the ferrite matrix and/or it may reflect load response associated with plastic deformation.
Table 4 shows the $DEC_{hkl}$ for all the alloys, the values are compared with values obtained from single crystal elastic constants calculations from references [47, 48].

Alloy composition showed an influence on the lattice strains of the ferrite phase. At the same level of applied stress, the high nickel alloy showed the largest lattice strains followed by the low nickel alloy, the manganese nickel alloy and finally the high manganese alloy. These results suggested that both nickel and manganese affected the lattice strains of the ferrite phase, while nickel increased the lattice strain, manganese decreased the lattice strain. Figure 8 shows the effect of alloy chemistry on the lattice strain of the ferrite BCC110 reflection. Similar trends were found for the BCC200 reflection. For all the alloys under study, the FCC111 and FCC311 lattice strains showed a similar behavior except that the stress at which plastic deformation started was lower for the high manganese alloy, which may be explained by the lower concentration of carbon in the austenite for this alloy (Figure 9).

Neutron diffraction experiments showed that the austenite in the low nickel alloy intercritically austenitized at 750°C and the high nickel alloy intercritical austenitized at 720°C did not undergo a transformation when loaded, which agrees with the XRD results. The austenite in the high manganese alloy intercritically austenitized at 750°C and 760°C started to transform to martensite before the applied stress reached the macroscopic yield strength of the materials. The manganese-nickel alloy intercritically austenitized at 750°C started to transform when the material reached the macroscopic yield strength. Neutron diffraction experiments did not show the transformation of the austenite in the low nickel and manganese nickel alloys intercritical austenitized at 770°C and the high nickel alloy intercritical austenitized at 740°C, in apparent contradiction with the XRD
results. However, these results were explained by the fact that the maximum stress reached on these samples during the neutron diffraction experiments was 500 MPa which was just above the yield strength of these alloys. Taking into account the x-ray and neutron diffraction results, it can be concluded that nickel alloyed austenite was more stable than manganese alloyed austenite. The austenite in the materials alloyed with nickel transformed after the material had reached the yield point or did not transform at all during deformation, while the austenite in the high manganese alloy transformed before the material had reached the yield point at very low strains. This behavior was attributed to the presence of large blocky austenite particles with low carbon content obtained in the high manganese IADI. Figure 10 shows the percentage of the initial austenite remaining as a function of applied stress obtained by neutron diffraction for some of the alloys and heat treatments conditions in this study that represent well the general behavior of each alloy. Values above one hundred percent indicated grain rotation during plastic deformation.

Conclusions

1. Austenite morphology and austenite carbon content were a function of alloy chemistry. High manganese concentration led to the formation of large blocky low carbon austenite, while austenite in nickel and manganese nickel alloyed IADI was present with a combination of plate-like austenite particles between bainitic ferrite (ausferrite) and some blocky austenite.

2. Tensile properties of IADI were a function of alloy chemistry and intercritical austenitizing temperature. Higher intercritical austenitizing temperatures increased ultimate and yield strength and decreased elongation. Manganese alloyed
IADI showed low elongation while nickel and nickel-manganese IADI showed high elongation. The results were explained by the differences in the morphology and chemistry of the austenite.

3. Lattice strains of ferrite were a function of alloy chemistry. Manganese decreased ferrite lattice strains while nickel increased ferrite lattice strains.

4. Austenite stability in IADI depended upon alloy chemistry. High manganese concentration produced untransformed austenite volumes of large blocky low carbon austenite particles that transformed to martensite at low strains. Austenite stability increased with the addition of nickel, which led to the transformation of the austenite after yielding or no transformation at all during deformation.

Acknowledgements

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Medellin Colombia; he is currently a PhD candidate at the University of Alabama at Bir-
mingham.
REFERENCES


Table 1. Chemical composition of the ductile iron alloys investigated.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Ni</th>
<th>Cu</th>
<th>Mo</th>
<th>Mg</th>
<th>S</th>
</tr>
</thead>
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<td>Low Ni</td>
<td>3.64</td>
<td>2.17</td>
<td>0.33</td>
<td>0.68</td>
<td>0.72</td>
<td>0.013</td>
<td>0.045</td>
<td>0.013</td>
</tr>
<tr>
<td>High Ni</td>
<td>3.92</td>
<td>2.34</td>
<td>0.33</td>
<td>2.47</td>
<td>0.75</td>
<td>0.012</td>
<td>0.040</td>
<td>0.016</td>
</tr>
<tr>
<td>High Mn</td>
<td>3.73</td>
<td>2.25</td>
<td>1.59</td>
<td>0.01</td>
<td>0.04</td>
<td>0.017</td>
<td>0.046</td>
<td>0.014</td>
</tr>
<tr>
<td>Mn-Ni</td>
<td>3.74</td>
<td>2.24</td>
<td>0.91</td>
<td>0.51</td>
<td>0.05</td>
<td>0.016</td>
<td>0.048</td>
<td>0.014</td>
</tr>
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Table 2. XRD austenite weight percent and estimated carbon content in the austenite.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Intercritical austenitizing temperature, °C</th>
<th>Unloaded/unstrained γ, wt%</th>
<th>Loaded/strained γ wt%</th>
<th>Remaining γ, %</th>
<th>C in γ, wt%</th>
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<tr>
<td>Low Ni</td>
<td>750</td>
<td>2.3</td>
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<tr>
<td></td>
<td>770</td>
<td>21.0</td>
<td>16.7</td>
<td>79.7</td>
<td>1.6</td>
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<tr>
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<td>1.0</td>
<td>1.1</td>
<td>NA</td>
<td>1.4</td>
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<tr>
<td></td>
<td>740</td>
<td>6.6</td>
<td>2.5</td>
<td>37.9</td>
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</tr>
<tr>
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<td>0.7</td>
<td>41.2</td>
<td>1.0</td>
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<tr>
<td></td>
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<td>5.1</td>
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<td>1.2</td>
</tr>
<tr>
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<td>0.2</td>
<td>20.0</td>
<td>1.2</td>
</tr>
<tr>
<td></td>
<td>770</td>
<td>12.7</td>
<td>6.5</td>
<td>51.0</td>
<td>1.5</td>
</tr>
</tbody>
</table>

Table 3. Tensile properties of IADI obtained.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Intercritical austenitizing temperature, °C</th>
<th>Austenite, wt%</th>
<th>UTS, MPa</th>
<th>YS0.2%offset, MPa</th>
<th>Elongation, %</th>
<th>Hardness, HBN</th>
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</thead>
<tbody>
<tr>
<td>Low Ni</td>
<td>750</td>
<td>2.3</td>
<td>516</td>
<td>347</td>
<td>18.0</td>
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<tr>
<td></td>
<td>770</td>
<td>21.0</td>
<td>777</td>
<td>498</td>
<td>16.0</td>
<td>255</td>
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<td>High Ni</td>
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<td>1.0</td>
<td>522</td>
<td>407</td>
<td>19.2</td>
<td>206</td>
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<tr>
<td></td>
<td>740</td>
<td>6.6</td>
<td>639</td>
<td>415</td>
<td>18.5</td>
<td>224</td>
</tr>
<tr>
<td>High Mn</td>
<td>750</td>
<td>1.7</td>
<td>587</td>
<td>324</td>
<td>6.5</td>
<td>208</td>
</tr>
<tr>
<td></td>
<td>760</td>
<td>6.1</td>
<td>725</td>
<td>403</td>
<td>4.0</td>
<td>265</td>
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<tr>
<td>Mn-Ni</td>
<td>750</td>
<td>1.0</td>
<td>482</td>
<td>298</td>
<td>19.0</td>
<td>184</td>
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<tr>
<td></td>
<td>770</td>
<td>12.7</td>
<td>704</td>
<td>432</td>
<td>14.7</td>
<td>251</td>
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Table 4. Diffraction elastic constants.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Intercritical austenitizing temperature, °C</th>
<th>Diffraction Elastic Constants (DEC$_{hk\ell}$), GPa</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>BCC110</td>
</tr>
<tr>
<td>Low Ni</td>
<td>750</td>
<td>195</td>
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<td></td>
<td>770</td>
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<td>198</td>
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<tr>
<td></td>
<td>740</td>
<td>205</td>
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<tr>
<td>High Mn</td>
<td>750</td>
<td>265</td>
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<tr>
<td></td>
<td>760</td>
<td>284</td>
</tr>
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<tr>
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<td>770</td>
<td>271</td>
</tr>
<tr>
<td>Calculated*</td>
<td>---</td>
<td>221</td>
</tr>
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</table>

nr: not reliable $R^2$ lower than 0.75.
* Based on single crystal elastic calculations for ferrite and austenite in references 47 and 48.


Figure 1. Dimensions of the Y-Block used to produce the ductile iron castings.
Figure 2. Representative optical microscope images of IADI (a) Low Ni alloy intercritically austenitized at 750°C for 4 hrs followed by 357°C for 2.25 hrs (b) Low Ni alloy intercritically austenitized at 770°C for 4 hrs followed by 357°C for 2.25 hrs. (c) High Mn alloy intercritically austenitized at 760°C for 4 hrs followed by 357°C for 2.25 hrs. (d) Mn-Ni alloy intercritically austenitized at 770°C for 4 hrs followed by 357°C for 2.25 hrs.
Figure 3. SEM secondary electron images of IADI. (a) Low Ni alloy intercritically austenitized at 750°C for 4 hrs followed by 357°C for 2.25 hrs (b) High Ni alloy intercritically austenitized at 740°C for 4 hrs followed by 357°C for 2.25 hrs. (c) High Mn alloy intercritically austenitized at 760°C for 4 hrs followed by 357°C for 2.25 hrs. (d) Mn-Ni alloy intercritically austenitized at 770°C for 4 hrs followed by 357°C for 2.25 hrs.
Figure 4. IADI ultimate tensile strength and yield strength as a function of ductile iron alloy, intercritical austenitizing temperature and initial austenite volume percent.

Figure 5. IADI elongation as a function of ductile iron alloy, intercritical austenitizing temperature and initial austenite volume percent.
Figure 6. Ultimate tensile strength vs elongation for ductile iron with different microstructures including the data obtained for IADI in this study.

Figure 7. Lattice strains in the direction parallel to the loading axis for the low nickel alloy.
Figure 8. Influence of alloy chemistry on the lattice strain of the BCC100 reflection.


Figure 9. Influence of alloy chemistry on the lattice strains of the austenite FCC111 reflection.

Figure 10. Amount of austenite remaining as a function of applied stress for some of the alloys and heat treatment conditions under study.
8. OVERALL CONCLUSIONS

Different aspects of intercritical heat treatments and steel were investigated in this dissertation. Three different studies were performed, which have provided new knowledge that will allow advances in the understanding and applications of this type of heat treatment. The general conclusions can be summarized as follows.

Intercritical heat treatments applied to common steel grades gave tensile properties comparable to those in DP and TRIP steels. These materials contained higher carbon and alloying concentrations, which provides better hardenability, allowing the future production of thicker parts than those fabricated with DP and TRIP steels. The intercritically austempered steel heat treatment may also be applied to casting alloys. Future development of the optimum heat treatment conditions, selection of the most promising alloys and detailed studies of hardenability in materials under an intercritical heat treatment are needed in order to widen the possibility of an industrial application.

Manganese-nickel alloyed ductile iron with a matrix of continuous free ferrite plus tempered martensite particles was successfully produced. This material exhibited tensile strengths in the range of pearlitic-ferritic grades with much larger elongations. The study demonstrated that during intercritical austenitizing, limited diffusion of substitutional alloying elements occurred as it does in the production of dual phase steels. Also, when a ductile iron with a pearlitic-ferrite matrix is intercritically austenitized, the carbon to form the austenite at high temperature comes mainly from the carbon available in the matrix and the diffusion of carbon from the graphite nodules is limited. Future develop-
ment of IAQ&TDI may include determining impact and fatigue properties of the material, evaluation of new alloys and hardenability studies to determine the thickness of the sections that can be successfully hardened. These studies will allow determination of specific applications for IAQ&TDI.

Studying the influence of manganese and nickel in IADI demonstrated that high manganese produced large, low carbon, blocky austenite located mainly in the intercellular boundaries that reduced the elongation and transformed to martensite during deformation at low strains. On the other hand, nickel produced a combination of blocky and plate-like austenite, with a good combination of elongation and strength. Also, nickel produced a more stable austenite that either transformed to martensite under deformation after yielding or did not transform at all. Nickel-manganese IADI showed good elongation in spite of the high manganese concentration (0.91 wt%), which has been found to be detrimental to the elongation in regular ADI at concentrations higher than 0.4 wt%. This finding indicates that higher manganese concentrations may be possible in the production of IADI, with promising improvements in the hardenability of the material. Future work on IADI may include finding the maximum manganese concentration in IADI that produces both high elongation and hardenability.
9. GENERAL REFERENCES


