INTERCRITICALLY AUSTENITIZED QUENCHED AND TEMPERED DUCTILE IRON

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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 percentages 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 400C (752F) and 500C (932F) for one hour. This paper reports the effects of volume percent martensite and tempering conditions on microstructure and tensile properties. Semi-quantitative chemistry and microhardness of ferrite and martensite were also determined as a function of 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 is limited. The results also showed that ultimate and yield strength and hardness increased and elongation decreased as the volume fraction of martensite increased. Tempering significantly increased the elongation with only a small decrease in the strengths.

Keywords: *intercritical austenitizing, tempering, ductile iron, dual phase*

Introduction

Heat treatment 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 230C (446F) and 400C (752F) and held for a 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 900C (1652F) for 480 seconds and then quenched in water. The results indicated that impact strength and elongation increased when tempering was performed at 400C-500C (752-932F) for 60 to 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 and 840C (1436 and 1544F) for 30 seconds and then quenched in oil at 100C (212F). After that, the samples were tempered at 550C (1022F) for 1 to 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 795-815C (1463-1499F) for 20 minutes followed by quenching in oil at 100C (212F). After that, the material was tempered at 500C (932F) for 1 to 5 hours. The results indicated that the variation of strength and ductility with tempering time are related to the intercritical 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 400C (752F) for one hour, and tempered at 500C (932F) 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-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 preheated 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 ~1520C (2768F) onto the treatment alloys. The treated iron was slagged-off, a chemistry sample taken and Y-blocks were poured at ~1450C (2642F). The Y-block molds were made from chemically bonded sand using 1.5% binder. Figure 1 illustrates 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 (0.52 in.) thick samples of the alloy in a preheated laboratory furnace [745-770C (1373F-1418F)], held 2.5 hours at temperature, and quenched in water. The samples were tempered at 200C (392F) 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 (0.52 x 0.98 in.), 165 mm (6.50 in.) long were intercritically austenitized at 750C (1382F), 760C (1400F) and 770C (1418F) for 2.5 hours and

Table 1. Chemical Composition of the Iron Used in Study

Element, wt%								
C	Si	Mn	Ni	Cu	Mg	S	P	Cr
3.87	2.33	0.69	0.50	0.19	0.05	0.016	0.019	0.04

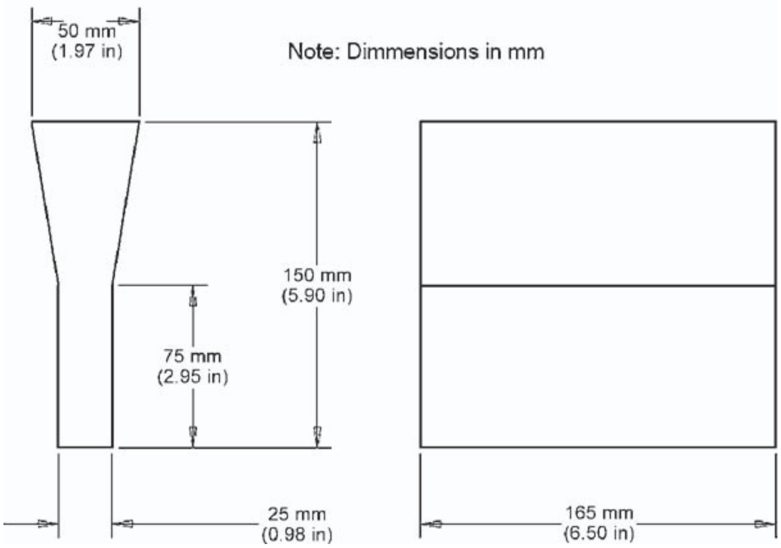

Figure 1. This illustration shows the dimensions (mm) of the Y-Block used to produce the ductile iron castings.

then quenched in a polymeric solution (UCON E 20%) at ambient temperature. Three different tempering conditions were evaluated: untempered, tempered at 400C (752F) for one hour, and tempered at 500C (932F) 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 dispersive 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 (0.35 x 1.42 in.) 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. A 10 mm (0.39 in.) diameter steel ball and a load of 3,000 kg (6,613.87 lb) 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 745C (1373F) to 37 vol% at 770C (1418F). 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 manganese 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 the segregation of silicon (which is a ferrite stabilizer) during the solidification process to the areas surrounding the graphite nodules.[3] X-ray diffraction (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 manganese 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.

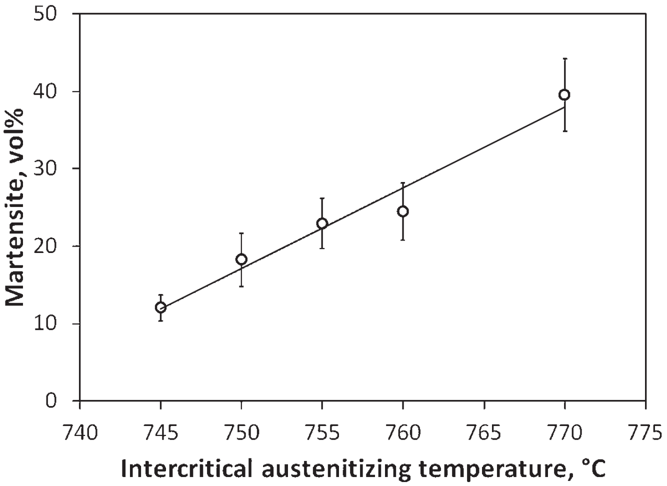

Figure 2. Volume percent of martensite as a function of intercritical austenitizing temperature for IAQ & TDI is shown graphically.

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] Previously 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

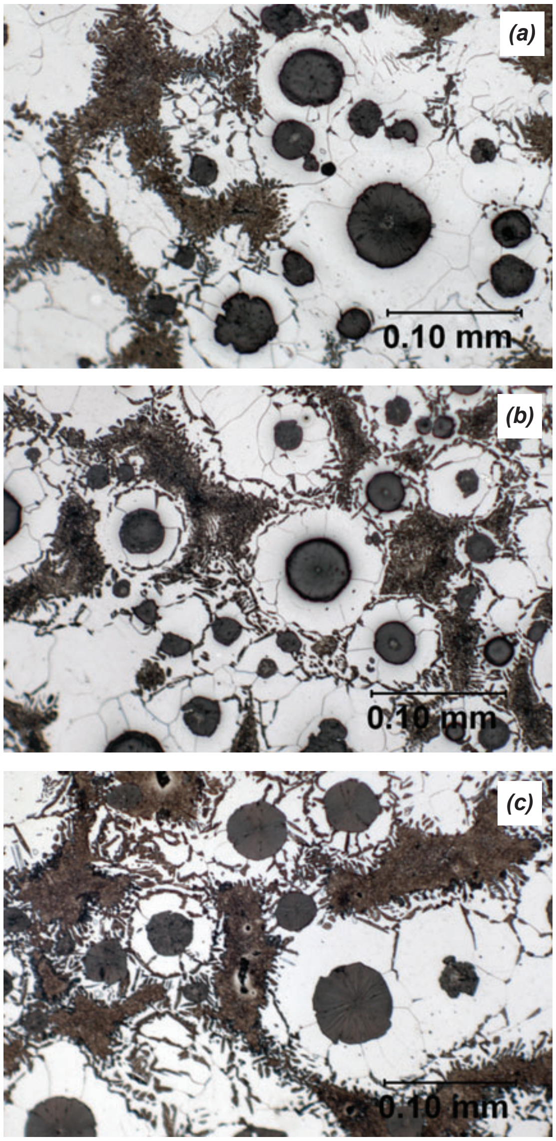

Figure 3. Representative microstructures of IAQ & TDI intercritically austenitized for 2.5 hours at (a) 750C (1382F)—16 vol.% martensite, (b) 760C (1400F)—24 vol.% martensite, and (c) 770C (1418F)—37 vol.% martensite.

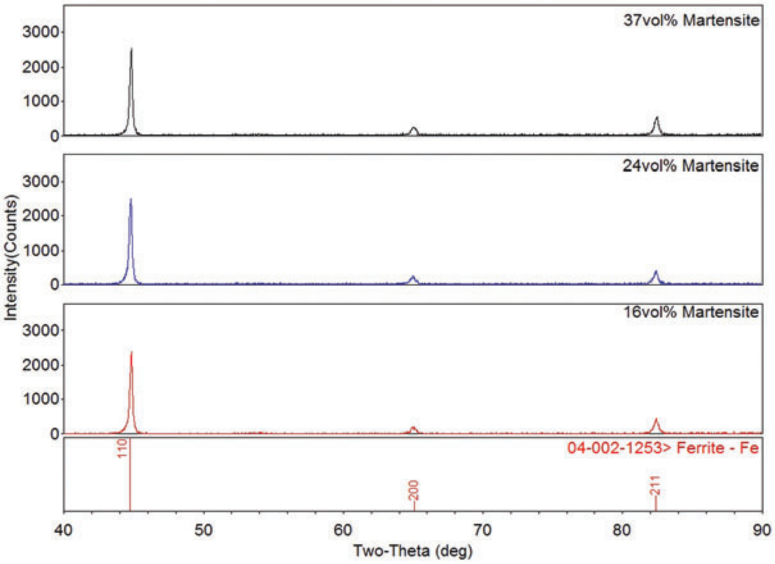

Figure 4. X-Ray Diffraction (XRD) spectrums of untempered IAQ & TDI with differing amounts of martensite.

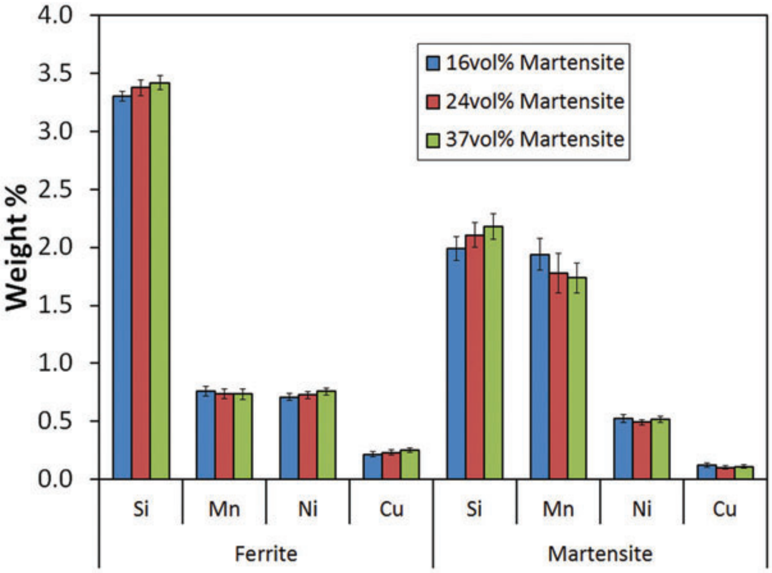

Figure 5. Semi-quantitative chemistry results for ferrite and martensite phases as a function of volume percent martensite are shown.

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 400C (752F) 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 500C (932F) 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 400C (752F)–500C (932F) increases elongation without affecting strength and that strength increases as volume fraction of martensite increases.[16-17]

The 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. The 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

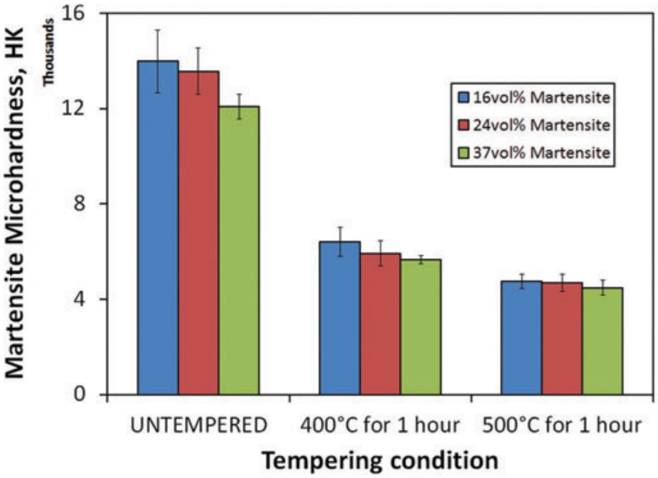

Figure 6. The effect of volume percent martensite and the tempering conditions on the microhardness of the martensite phase is shown.

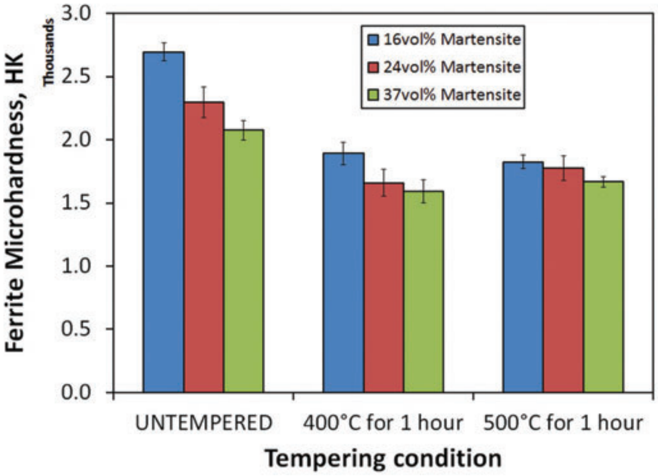

Figure 7. The effect of volume percent martensite and the tempering conditions on the microhardness of the ferrite phase is shown.

Table 2. Semi-quantitative Chemistry Analysis Results in wt % Ferrite and Martensite

Phase	Element	Martensite, vol%		
		16	24	37
Ferrite	Si	3.30 ± 0.04	3.38 ± 0.07	3.42 ± 0.06
	Mn	0.76 ± 0.04	0.74 ± 0.04	0.73 ± 0.05
	Ni	0.71 ± 0.03	0.73 ± 0.03	0.76 ± 0.03
	Cu	0.22 ± 0.02	0.23 ± 0.02	0.25 ± 0.02
Martensite	Si	1.99 ± 0.1	2.10 ± 0.11	2.18 ± 0.11
	Mn	1.94 ± 0.14	1.78 ± 0.17	1.74 ± 0.13
	Ni	0.53 ± 0.03	0.49 ± 0.03	0.52 ± 0.03
	Cu	0.13 ± 0.02	0.10 ± 0.02	0.11 ± 0.01

Table 3. Mechanical Properties of IAQ & TDI

Austenitizing Temperature, °C/F	Martensite, vol%	Tempering Condition	UTS, MPa	YS, MPa	Elongation, %	HBN
750/1382	16	Untempered	532	329	7.4	197
		400C(752F) 1 hr	524	325	14.3	187
		500C(932F) 1hr	513	327	15.5	175
760/1400	24	Untempered	605	353	5.8	216
		400C(752F) 1 hr	609	363	10.3	209
		500C(932F) 1hr	585	371	11.8	195
770/1418	37	Untempered	643	376	4.9	241
		400C(752F) 1 hr	677	406	8.5	226
		500C(932F) 1hr	600	377	12.0	204

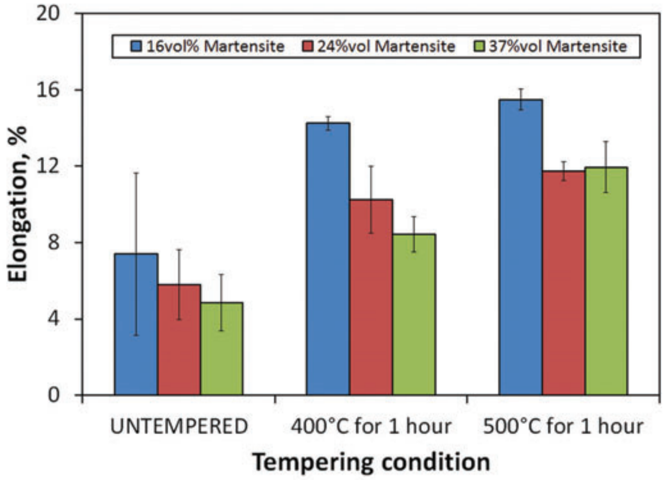

Figure 8. The effect of volume percent martensite and the tempering conditions on the elongation of IAQ & TDI is shown.

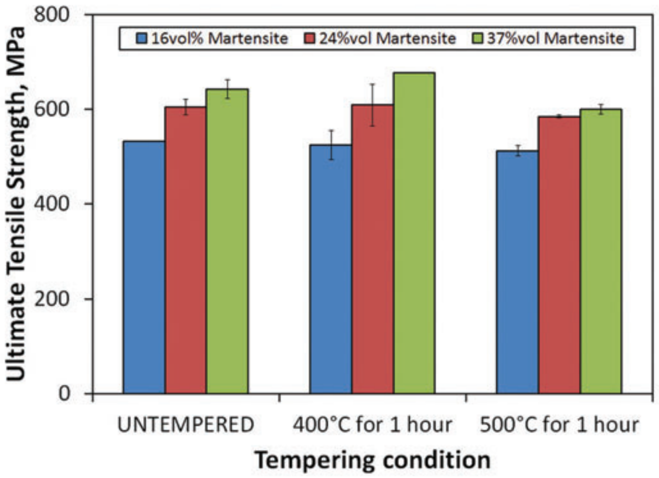

Figure 9. The effect of volume percent martensite and the tempering conditions on the ultimate tensile strength of IAQ & TDI is shown.

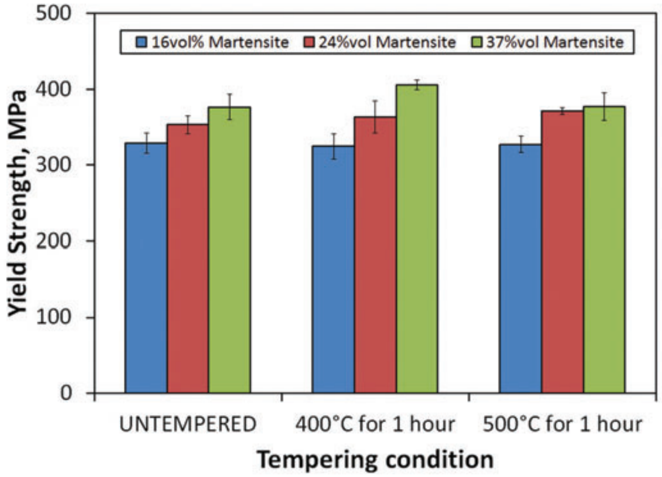

Figure 10. The effect of volume percent martensite and the tempering conditions on yield strength of IAQ & TDI is shown.

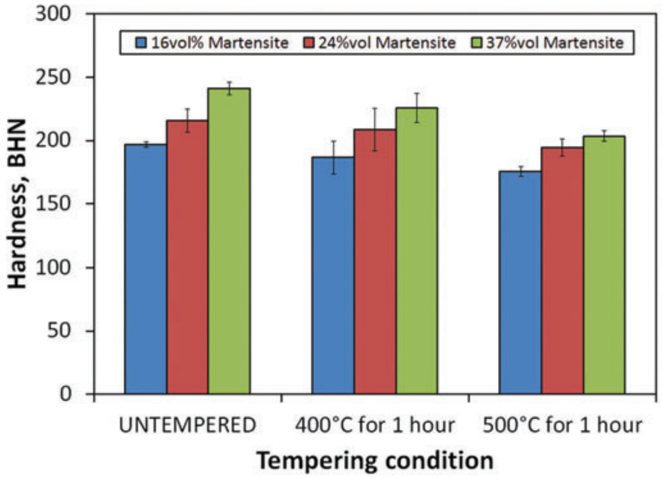

Figure 11. The effect of volume percent martensite and the tempering conditions on the hardness of IAQ & TDI is shown.

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 elongation and ultimate tensile strength for ductile iron with different microstructures,[22] including the data obtained for IAQ & TDI in this study.

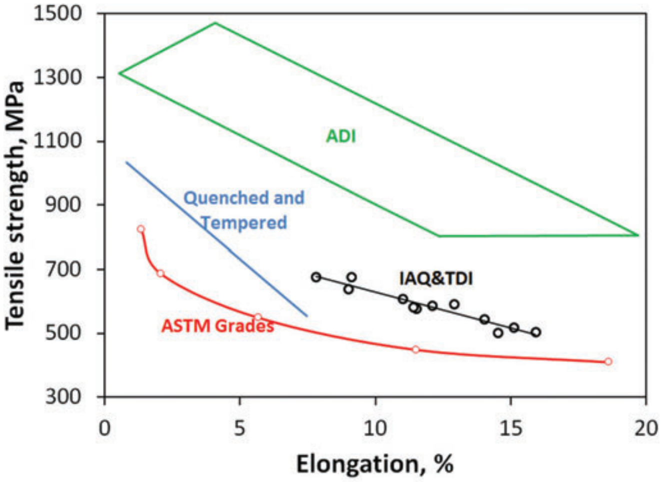

Figure 12. The relationship between elongation and ultimate tensile strength for ductile iron with different microstructures is shown graphically.

Conclusions

- The concentrations of silicon, manganese, nickel and copper in the martensite and ferrite were determined by the segregation of these elements during solidification of the alloy rather than by diffusion during the intercritical austenitizing step of the heat treatment.
- 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 the matrix carbon rather than the nodules was the primary carbon source in the martensite.
- 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.
- 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.
- The IAQ & TDI had ultimate tensile strengths and yield strengths similar to pearlitic ductile irons but had higher elongations (2.5 to 3.7 times higher). These properties are provided by 1.) a microstructure of well-formed graphite nodules in a matrix of ferrite plus particles of tempered martensite and 2.) the solid solution strengthening of manganese and nickel used as alloying elements.

Acknowledgements

This work is partially a result of a project developed at the UAB (University of Alabama-Birmingham, AL, USA) Materials Engineering Course MSE 409/509 Principles of Metal Casting, under the supervision of Dr. Alan P. Druschitz.

The authors would like to thank John Griffin and the UAB casting group for assistance in preparing the cast alloy, Dr. Gregg Janowski for his exhaustive review of the manuscript and his help interpreting the x-ray diffraction (XRD) information and Grede-Columbiana, Columbiana, AL (USA) and Tom Hewer at Webb Wheel, Cullman, AL (USA) for performing the chemical analysis.

Research was sponsored by the U.S. Dept. of the Army ARDEC (Armament Research, Development and Engineering Center)-Benet Laboratories and managed by AFS (American Foundry Society) on behalf of the U.S. Army Contracting Command Joint Munitions & Lethality Contracting Center and was accomplished under Cooperative Agreement Number W15QKN-11-2-0001.

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