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Influence of martensite volume fraction and tempering time on tensile properties of partially austenitized in the ($\alpha + \gamma$) temperature range and quenched + tempered ferritic ductile iron

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In the present study, the effect of martensite volume fraction and tempering time on the tensile properties of ferritic ductile iron with dual matrix structure was investigated. For this purpose, specimens were intercritically annealed (partially austenitized) in the two phase region ($\alpha + \gamma$) at various temperatures of 795 and 815°C for 20 min and then quenched into oil held at 100°C to obtain different martensite volume fractions. Some specimens were also conventionally heat treated (austenitized at 900°C and then quenched + tempered) for a comparison reason. The results showed that a structure having proeutectoid ferrite plus martensite has been developed and volume fraction of proeutectoid ferrite and martensite can be controlled to influence the strength and ductility. Specimens quenched from the ($\alpha + \gamma$) temperature range exhibited much greater ductility than conventionally heat treated specimens. The tensile strength increased and ductility decreased with increasing martensite content. By increasing the tempering time, the yield and UTS decreased and ductility increased. The specimens tempered for 3 h and having 62% martensite volume fraction (MVF) exhibited the best combination of high strength and ductility. The tensile and proof stress of this material is much higher than pearlitic grades and ductility is lower than ferritic grades. The specimen tempered for 3 h and having ~25% MVF exhibited the best combination of high strength and ductility compared to ferritic grades. However its strength is slightly lower but the ductility is almost three times higher than pearlitic grades.

1. Introduction

The mechanical properties of ductile irons are controlled primarily by their matrix structure. Therefore, modification in the amount or distribution of matrix phases or microstructures can modify mechanical properties.

In the newly developed ductile cast iron with dual matrix structure, the structure consists of ferrite, and martensite or ausferrite (bainitic ferrite and austenite). Therefore it is also called Dual Matrix Structure (DMS).

So far a few attempts have been made to clarify the mechanical properties of DMS with ferrite + martensite structure [1–4]. In those studies DMS were obtained by rapidly heating ductile iron (DI) into the single phase (γ) region and then holding the material at this temperature for a very short time (generally less then 1 min) and then quenching the material before the growth of austenite into surrounding ferrite is completed. The shortcoming of this heat treatment procedure is the control martensite volume fraction (MVF). No efforts have so far been made to obtain DMS by intercritical annealing in the two phase region ($\alpha + \gamma$) and to optimize the mechanical properties of DMS by controlling the proeutectoid ferrite and the MVF.

Silicon is inherently present in DI and it has the effect of modifying the Fe-C phase diagram, a three-phase region of ($\alpha + \gamma$ + graphite) is introduced into the Fe-C-Si phase diagram [5]. Consequently austenitising at low solution treatment temperatures the intercritical annealing temperature (ICAT) ranges produces structures containing proeutectoid ferrite [1, 6–9].

Control over the ICAT can play an important role in controlling austenite volume fraction and its carbon content. Decreasing the austenitizing temperature decreases the austenite volume fraction and its carbon content as predicted by the lever rule. This important feature can provide advantage of controlling

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TABLE I Chemical composition of unalloyed ductile iron used (wt%)

C	Si	Mn	P	S	Cr	Mo	Ni	Al	Co
3.50	2.630	0.318	0.0190	0.009	0.031	0.0421	0.0423	0.003	<0.001
Cu	**Nb**	**Ti**	**V**	**W**	**Pb**	**Sn**	**Mg**	**Sb**	**Fe**
0.0552	<0.002	<0.0120	<0.001	0.005	<0.0002	0.0058	0.0471	0.0055	Rest

proeutectoid ferrite, and austenite contents during annealing in the two-phase region.

In the current study, DI with DMS having proeutectoid ferrite, and martensite structure were obtained by the intercritical annealing (partially austenitizing) the ductile iron in two-phase region ($\alpha + \gamma$) and then quenching rapidly to room temperature. The ICAT range indicated here corresponds to the ($\alpha + \gamma +$ graphite) region. In this article, this is indicated in short hand as the ($\alpha + \gamma$) temperature range. The intercritical annealing temperature introduces proeutectoid ferrite into the austenitized structure producing a proeutectoid ferrite and martensite matrix after quenching.

Most of the studies on DMS with martensite have been carried out on ductile iron with a predominantly pearlitic microstructure [1–4]. However, no information is currently available in literature on the mechanical properties of DMS with ferrite + martensite produced from unalloyed ferritic ductile iron.

The purpose of the present study is quantify these effects and optimize tempering time for an unalloyed ferritic cast iron containing 3.5 C wt%, 2.63 Si wt% and 0.318 Mn wt%. The influence of MVF and tempering time on mechanical properties are reported.

2. Experimental procedure

The chemical composition and microstructure of the as cast unalloyed ferritic DI used in the present study is given in Table I. The DI was produced in a medium frequency induction furnace in a commercial foundry. The tundish cover ladle method was used to treat a 250 kg melt of iron with 6–7% Mg containing ferrosilicon alloy at 1450°C. Final inoculation was carried out with a 75% ferrosilicon alloy. The melt at the temperature between 1450 and 1400°C was cast into Y block sand mould, which was prepared in accordance with ISO 1083.

The preliminary investigation was to determine the dependence of austenite (martensite at room temperature) volume fraction on ICAT. For this purposes samples 10 × 10 × 5 mm thick machined from the bottom section of the Y-block were annealed for 20 min in a normal atmosphere at a series of temperatures from 780 to 840°C for Ae_1 and upper critical temperature respectively (Fig. 1) and then quenched into oil held at 100°C. Ae_1 temperature limits were predicted from the empirical formulae of Andrews [10]. The austenite formed during intercritical annealing was assumed to transform into martensite. The MVF was determined by point counting on metallographic sections etched in 2% nital. As a result of this preliminary study, two ICAT of 795, and 815°C (These temperatures corresponds to ~25%, and ~62% MVF) (see Fig. 1) were selected for a detailed study of the development of the DMS with different MVF. The as cast material had ferrite + graphite structure (Fig. 2). This microstructure was labeled as "A" for further reference. The microstructure of specimens A was the starting point for subsequent DMS heat treatment. As cast samples were also heat-treated at the conventional austenitizing temperature of 900°C in single-phase region (γ) and then quenched into oil for comparison.

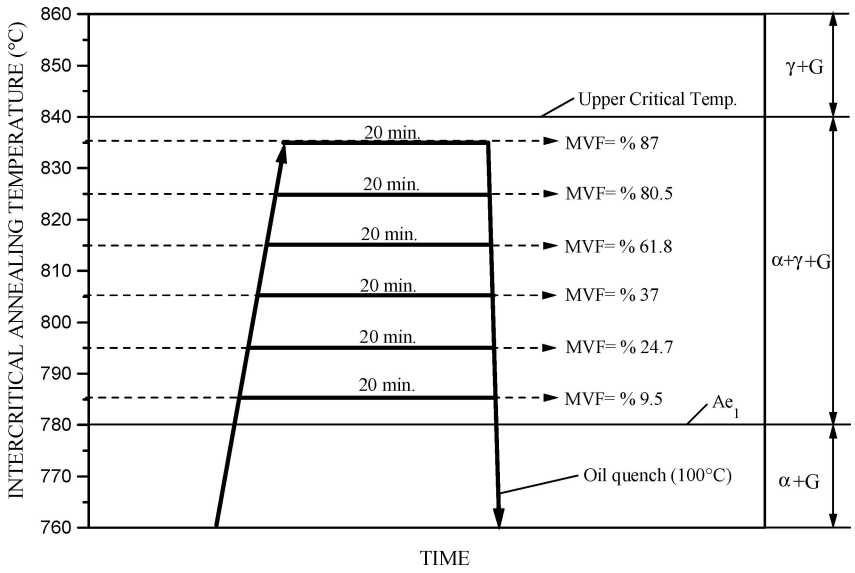

Figure 1 Dependence of austenite (martensite) content on intercritical annealing temperature.

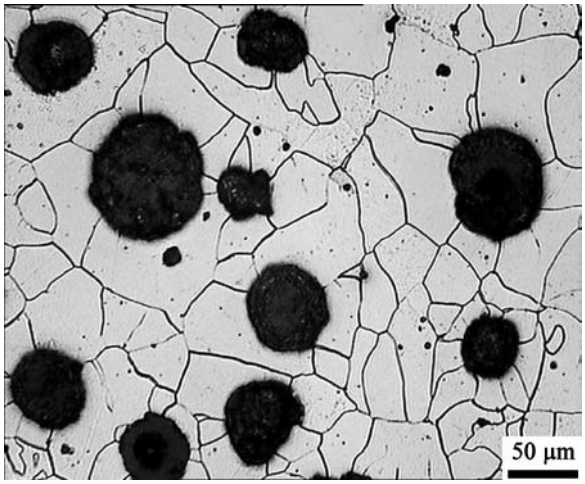

Figure 2 Microstructure of as-cast ductile iron. Etchant: 2% Nital.

Based on the results of the preliminary investigation, specimens 10 × 10 × 5 mm machined from the bottom section of Y block were intercritically annealed at ICAT of 795, and 815°C for 20 min. The specimens were then quenched into oil held 100°C to produce DMS with different MVF. The quenched samples were tempered at 500°C for various times from 1 to 5 h. Fig. 3 provides a summary of heat treatments. Throughout these heat treatments, the temperature of each specimen was monitored by a thermocouple spot-welded to the center of one of its faces.

The specimens were coded according to ICAT, starting microstructure and tempering time. For example, in specimen code 815AT3, 815 stands for ICAT, A for starting microstructure and T3 for 3 h tempering time. On the other hand the conventionally heat-treated sample is coded as B900. The proportions of the constituents present were determined by point counting on etched (Nital) metallographic sections. Between 1000 and 2000 points were counted to keep the standard error of the volume fraction of phases below 6%. The nodule count was 195 mm^{-2}.

The proportions of phases determined by point counting are listed in Table II. Following the microstructural studies, tensile test specimens were machined from the bottom section of Y block (Fig. 4). Tensile specimens were machined after heat treatments to remove any decarburized layer. Metallographic observations and quantitative measurement showed that similar microstructures produced in 5 mm thick specimens for each series were also produced in the corresponding tensile test specimens.

Tension tests were carried out at room temperature in ambient air using a DARTEC machine with 60 kN loading capacity at a cross-head speed of 1 mm/min. An extensometer set to a gauge length of 10 mm was used for strain measurement. An automatic record of load versus percent elongation was made. At least five specimens were tensile tested for each heat treatment conditions and average values were calculated.

Hardness measurements were made using an Instron Wilson Tukan 2100 Version 1–36,7 hardness-testing machine. Several specimens were evaluated for each condition. At least five indents were made at each location and average values were taken.

3. Results and discussion

3.1. Heat treatments and microstructures

Fig. 5 is an illustration showing the locations on the Fe-C-Si phase diagram of the conventional heat treatment and the production of DMS with various MVF.

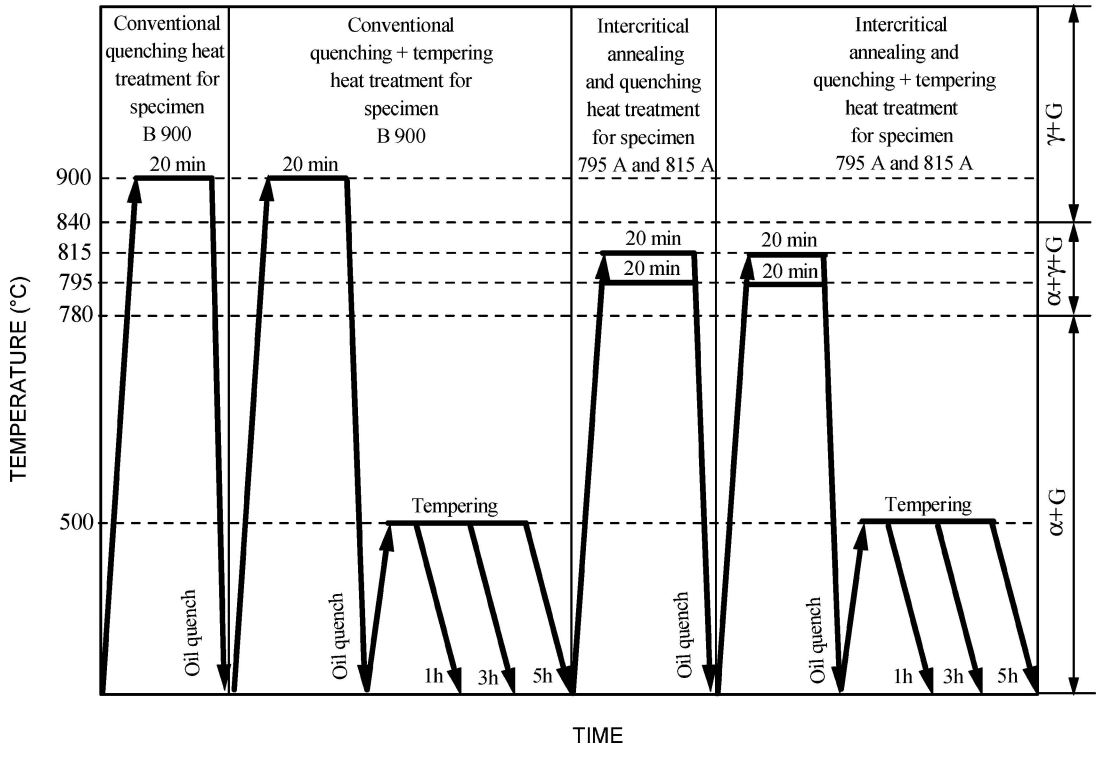

Figure 3 Summary of heat treatments.

TABLE II Results of metallographic measurement and tensile properties (Average values ±3%)

Spec. code	ICAT (°C)	Temp. time (hr)	Marten. content vol-%	Proeutec ferrite content vol-%	0.2% proof strengt. (MPa)	True tensile strengt. (MPa)	True uniform elong. (%)	Total elong. (%)	Reduc. in area (%)
As cast	–	–	–	89.8	261.5	465.2	16	27.7	26.6
795A	795	–	24.7	65	411	578.2	8.7	9.2	3.9
795AT1	795	1	24.7	65	261	467.3	14.3	21.7	21
795AT3	795	3	24.7	65	239	395.4	15.7	17	13.2
795AT5	795	5	24.7	65	245	400.5	13.4	14.6	11.6
815A			61.8	28	353	600.8	5.5	6.3	5.5
815AT1	815	1	61.8	28	368	670.4	8.9	10.9	8.8
815AT3	815	3	61.8	28	362	580.3	10.8	13.2	10
815AT5	815	5	61.8	28	368	562.7	9.7	12.5	11.9
B900			89.7	-	1121	1341.2	1.1	1.1	1.3
B900T1		1	89.7	-	933	1227.2	2	2.1	2.9
B900T3		3	89.7	-	900	1097.6	2.4	2.4	2.6
B900T5		5	89.7	-	832	1061.4	2.8	2.8	2.9

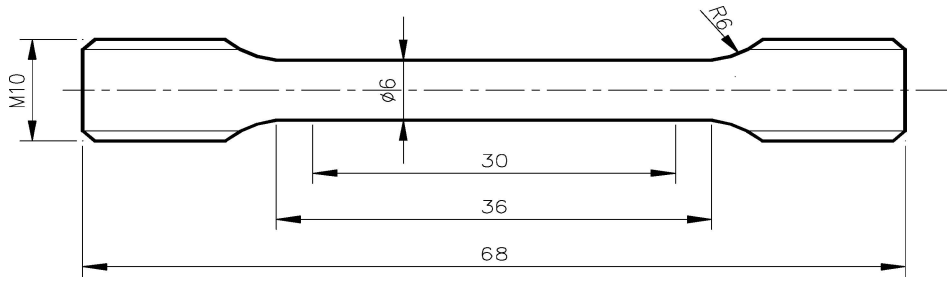

Figure 4 Dimension of tensile test specimen (in mm).

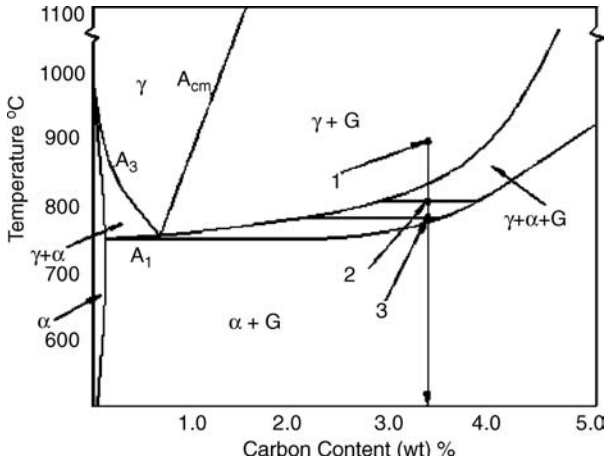

Figure 5 Schematic illustration of Fe-Fe_3C phase diagram and the production of dual matrix structure with various AFVF in the ductile cast iron (Phase volume fractions obtained experimentally are not represented exactly because true equilibrium was not reached during the intercritical heat treatment).

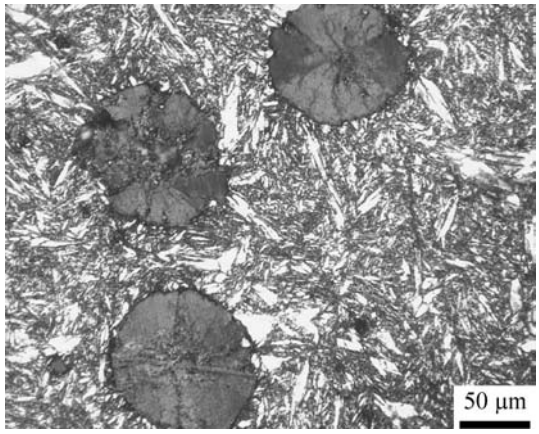

Figure 6 Microstructure of quenched sample from 900°C. Etchant: 2% nital.

Conventional heat treatment involves heating the ductile iron up to 875–925°C (in this study 900°C) and then quenching and tempering. During the conventional heat treatment at the temperature of ~900°C (points 1), the specimen remains in austenitic single-phase region. The conventional heat treatment of the B900 specimen produced a typical martensitic structure throughout the specimen (Fig. 6).

On the other hand at the ICAT of 815°C (point 2) or 795°C (point 3), the specimen is in the austenite + ferrite region ($\alpha + \gamma$). On heating ferritic microstructure A (as cast) to the ICAT, austenite nucleated at prior ferrite/ferrite grain boundaries which are located in the eutectic cell (Fig. 7a) and then grew into the ferrite (Fig. 7b). Phase volume fractions obtained experimentally are not represented exactly because true equilibrium was not reached during the intercritical heat treatment for 20 min.

The quenching of samples from different ICAT into oil held 100°C produced DMS with different MVF which was either restricted to eutectic cell boundaries and a continuous network or an isolated martensitic structure along the eutectic cell boundary formed depending on MVF (Figs 7b and 8).

Some alloying elements like silicon and manganese are inherently present in ductile iron. Mn segregates in the eutectic cell while Si segregates near the graphite

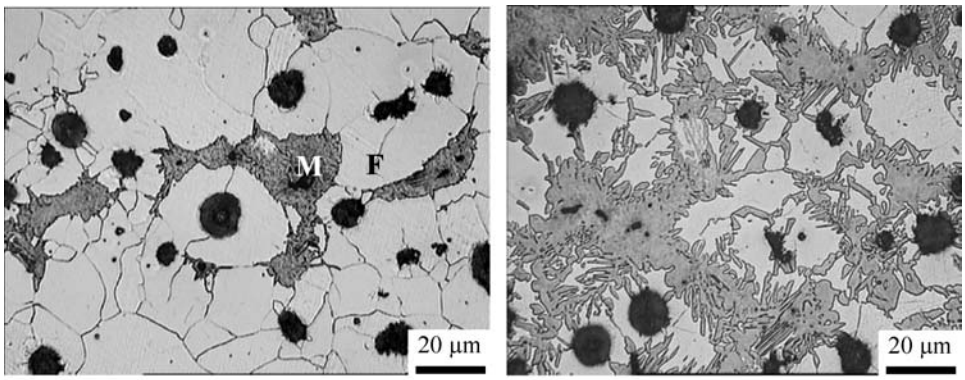

Figure 7 Micrographs of specimen 815A partially austenitized at the intercritical temperature of 815°C for 1 min (a) and 20 min (b) and then oil quenched. Etchant: 2% nital.

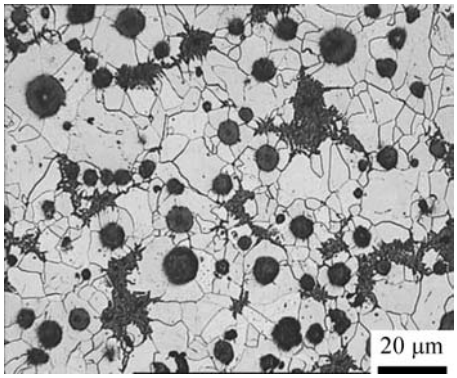

Figure 8 Micrographs of specimen 795A partially austenitized at the intercritical temperature of 795°C for 20 min and then oil quenched. Etchant: 2% nital.

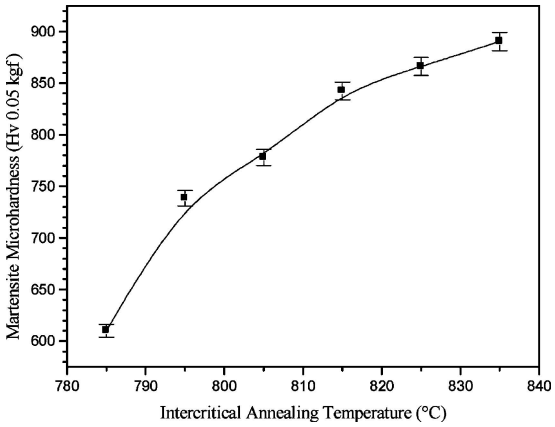

Figure 9 The relationship between ICAT and martensite microhardness of specimens reflecting carbon content of martensite.

nodule. Mn reduces the activity of carbon and encourages austenite nucleation (an austenite stabiliser) while Si increases the activity of carbon (a ferrite stabiliser). Consequently, since Mn segregates in the eutectic cell and only the minimum volume diffusion of carbon is needed for the nucleation of austenite, it is reasonable to assume that the eutectic cell is the most potent site for austenite nucleation (5). The reason for the absence of formation of austenite near graphite nodules may be attributable to the effect of Si segregation on the reduction of the austenite formation rate [6].

Another facet of ICAT heat treatment is that in the austenite+ferrite region, the austenite volume fraction and its carbon content depends on the ICAT. When the ICAT increases from points 3 to 2 (Fig. 5), the austenite volume fraction and its carbon content increases and proeutectoid ferrite volume fraction (PFVF) decreases as defined by the lever rule. This means that the MVF and PFVF can be controlled by using this heat treatment as parent austenite formed during intercritical annealing transforms into martensite upon quenching. The microhardness variation in martensite with ICAT in quenched samples from ICAT range is given in Fig. 9. Variation in the microhardness of martensite with ICAT is a good indication of the martensite carbon content due to the diffusionless nature of martensite transformation from austenite.

3.2. Tensile properties

3.2.1. 0.2% proof and tensile strength

The average values of the 0.2% proof and tensile strength, uniform and total elongation obtained for each the heat treatment conditions investigated are given in Table II.

In the quenched samples from ICAT range, it is readily apparent that both the 0.2% proof and tensile strength increased with increasing MVF or with decreasing PFVF (Fig. 10 and Table II). This result is in agreement with much of the existing literature,

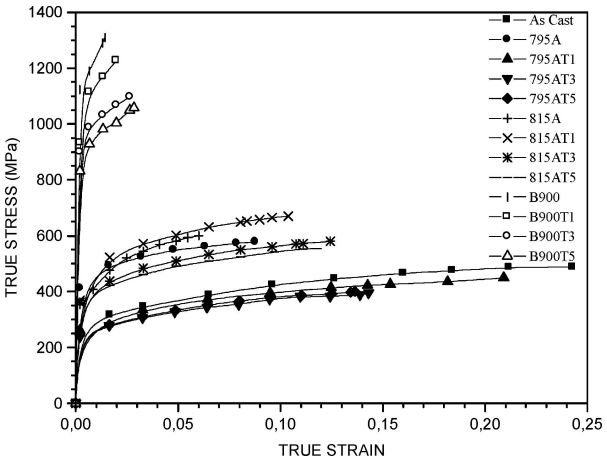

Figure 10 True stress-strain curves for specimens.

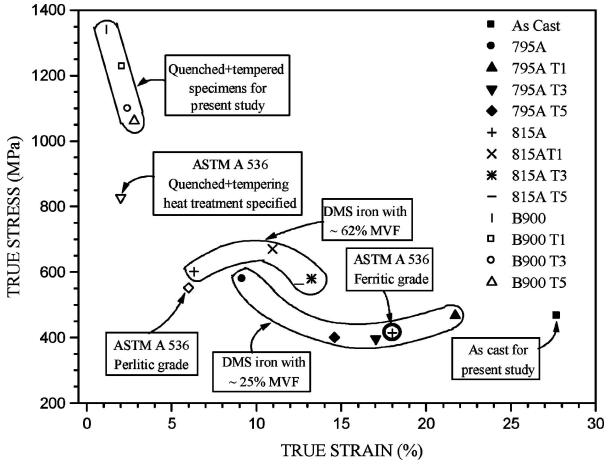

Figure 11 The comparison of the engineering tensile strength and ductility of present study specimens with quenched+tempered, pearlitic and ferritic grades.

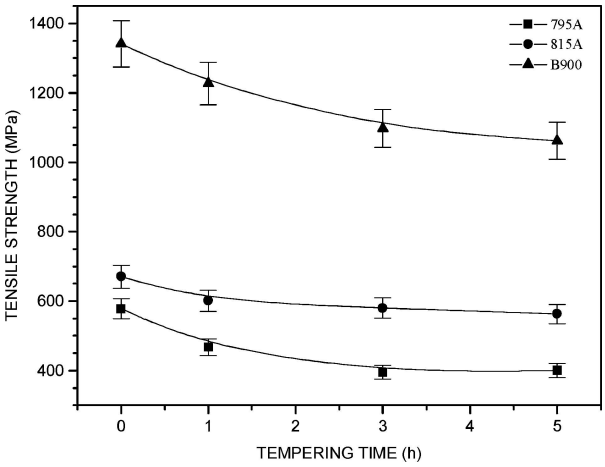

Figure 13 The relationship between tempering time tensile strength of various specimens.

which indicates an approximately linear relationship with MVF [11–14]. The conventionally heat treated material (B900), with its wholly martensitic structure throughout the specimen, had the highest 0.2% proof and tensile strength and the lowest elongation values among the different austenitizing temperatures.

The 815AT1 specimen tempered for 3 h and having ∼62% MVF exhibited the best combination of high strength and ductility. The ductility and strength of this material is much higher than pearlitic grades and ductility is lower than ferritic grades (Fig. 11). On the other hand, the 795AT1 specimen tempered for 1 h and having ∼25% MVF exhibited the best combination of high strength and ductility compared to ferritic grades. However its strength is slightly lower but ductility almost more than three times higher than pearlitic grades (Fig. 11).

Figs 12 and 13 shows that increasing the tempering period for up to 3 h reduces proof and tensile strength of A815 and A795 specimens, and thereafter, they both levels off. The examination of the strength-tempering period curves reveals similar behavior in steel (15,16). However the proof and tensile strength of specimen B900, decreases regularly up to 5 h tempering time. This result may arise from the carbon content of martensite depending on austenitizing temperature (Fig. 9).

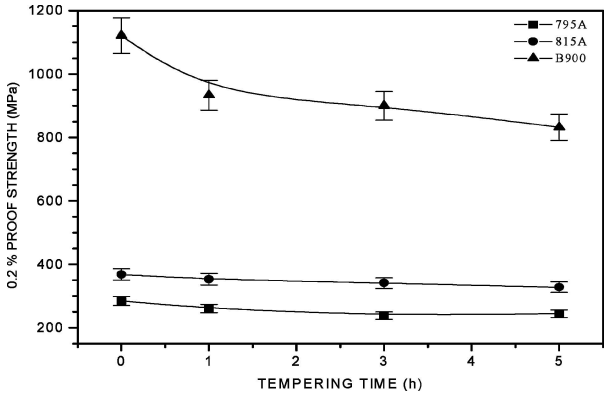

Figure 12 The relationship between tempering time and 0.2% proof strength of various specimens.

3.2.2. Uniform and total elongation

With respect to ductility, the lowest values of uniform and total elongation were recorded with conventionally heat-treated B900 material. Within each of the series of specimens quenched from ICAT, both uniform and total elongation increased with increasing PFVF (Fig. 10 and Table II).

Quenching from the ICAT range was very effective for improving the ductility of DI with DMS. The ductility is very sensitive to PFVF. Proeutectoid ferrite increases the elongation significantly. The lower strength reduces the area under the stress-strain curve in the presence of ferrite as shown in Fig. 10 and Table II.

In series A specimens, the peak elongation occur as the ICAT is decreased to 795°C. The results demonstrate that the introduction of proeutectoid ferrite is a further means of adjusting the mechanical properties of DI with DMS.

The degree of continuity of martensite structure network along intercellular boundary could be an important factor in determining ferrite deformation degree around graphite nodules. As mentioned before in this study quenching from different ICAT produced martensitic structure with different MVF restricted to eutectic cell boundaries and an isolated, and continuous network of martensitic structure along the intercellular boundary depending on MVF. In the specimen with higher MVF, ferrite around graphite nodules is completely surrounded by martensite structure as shown in Fig. 7b. In such a microstructure, high strength martensite structure may restrict deformation of a larger fraction of the total volume of the low strength ferrite under tensile loading. Therefore the ductility decreases with increasing continuity of martensite structure along eutectic cell boundaries.

Particularly the total and uniform elongation of the A795 specimen was superior to that of specimen B900 with nearly wholly martensitic structure throughout the specimen produced by conventional heat treatment.

For specimens 815A experimental results related to the effect of tempering period (see Figs 14 and 15) show that increasing the tempering period increases the uniform and total elongation percentage for tempering

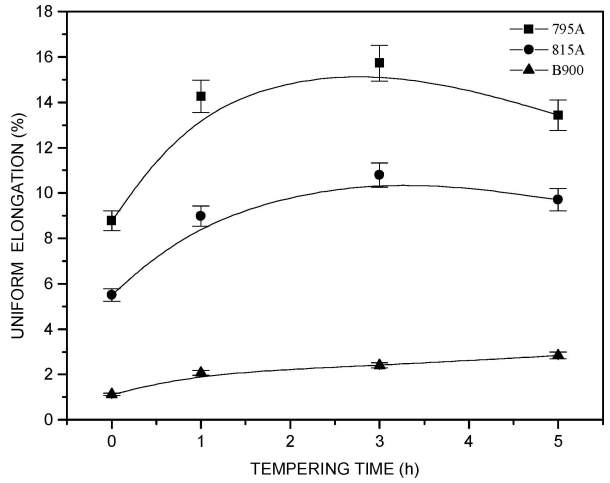

Figure 14 The relationship between tempering time and uniform elongation of various specimens.

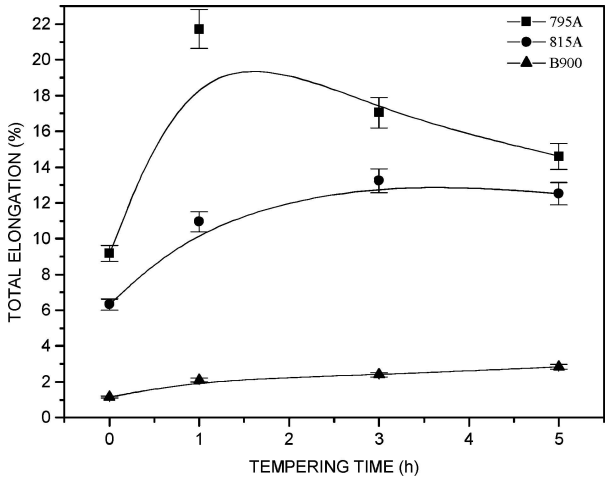

Figure 15 The relationship between tempering time and total elongation of various specimens.

periods up to 3 h followed by a gradual decrease. However in specimen 795A the total elongation increases sharply up to 1 h tempering time thereafter it decreases sharply. This result is similar to the strength-tempering of steel with low carbon [15, 16].

For specimen B900 tempering series, the gradual increase in both uniform and total elongation is continuous with increasing tempering time. This result is also attributable to the martensite carbon content of this specimen (Fig. 9).

As cast samples with a ferrite matrix exhibited highest ductility and generally lower tensile strength than all the heat-treated conditions tested.

4. Conclusions

1. Ductile iron with dual matrix structure exhibits much greater ductility than conventionally quenched + tempered ductile iron.

2. Proeutectoid ferrite volume fraction and martensite volume fraction can be controlled to influence the strength and ductility of ductile iron with dual matrix structure.

3. For any combination of martensite volume fraction and tempering period and for tempering periods of up to 5 h, the amount of tensile strength and ductility can satisfactorily be optimized.

4. The variation in both strength and ductility with tempering time is dependent on austenitizing or intercritical annealing temperature or carbon content of martensite.

5. The specimens tempered for 3 h and having ∼62% martensite volume fraction exhibited the best combination of high strength and ductility. The proof and tensile strength of this material is much higher than pearlitic grades and ductility is lower than ferritic grades.

6. The specimen tempered for 3 h and having ∼25% martensite volume fraction exhibited the best combination of high strength and ductility compared to ferritic grades. However its strength is slightly lower but ductility three times higher than pearlitic grades.

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