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High Silicon Ductile Iron: Possible Uses in the Production of Parts with "Dual Phase ADI" Microstructure

Alejandro BASSO,[1)]* Martín CALDERA,[1)] Graciela RIVERA[1)] and Jorge SIKORA[2)]

1) Graduate Student, Faculty of Engineering, INTEMA, National University of Mar del Plata, CONICET, Av. Juan B, Justo 4302, (B7608FDQ) Mar del Plata, ARGENTINA. E-mail: abasso@fi.mdp.edu.ar, mcaldera@fi.mdp.edu.ar, glrivera@fi.mdp.edu.ar
2) Faculty of Engineering, INTEMA, National University of Mar del Plata, CONICET, Av. Juan B, Justo 4302, (B7608FDQ) Mar del Plata, ARGENTINA. Passed away in 2011.

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The aim of this paper is to study the advantages and disadvantages of using high silicon ductile iron to produce cast parts with "Dual Phase ADI" microstructure. In this study, four ductile irons with different percentages of silicon: 2.4, 3.1, 3.5, and 4.2%, respectively, were used. Samples from each cast were subjected to heat treatment in order to determine the intercritical interval. Significant increase in the upper and lower critical temperatures was found as the amount of silicon incremented. However, the difference between the lower and upper critical temperatures (intercritical interval amplitude) remained nearly constant for all ductile irons.

The materials were metallographically and mechanically characterized in the as-cast conditions and after full annealing heat treatments. Tensile, hardness and impact properties were determined as a function of the silicon level.

The results suggest that high silicon ductile iron (Si content ranging from 3.0 to 3.6%) can be used in the manufacture of cast parts with "Dual Phase ADI" microstructures, yielding undeniable advantages in the production process if compared to low and medium silicon ductile irons.

KEY WORDS: silicon; ductile iron; Dual Phase ADI; microstructure; mechanical properties.

1. Introduccion

It is generally recognized that ductile iron (DI) is being increasingly applied to high mechanical performance parts manufacturing, replacing cast and forged steels. Despite its use in a wide number of applications, generally speaking, DI is not commonly utilized in mechanical components requiring very high mechanical strength, ductility and toughness. In said cases, steels continue to be the alternative of choice. Notwithstanding this, in the last years, a new type of DI called "Dual Phase ADI" has been developed, which has substantially improved mechanical properties. This improvement is explained by the novelty of this DI microstructure, which is composed of different amounts of free (or allotriomorphic) ferrite and ausferrite (ADI regular structure).[1–11)]

Special thermal cycles have been developed by different researchers in order to obtain Dual Phase ADI. One of them has been advanced and used by Kilicli *et al.*[7,8)] and Basso *et al.*[4–6)] It consists in subjecting fully ferritic DI parts to an incomplete austenitization stage, at different temperatures within the intercritical interval of the Fe–C–Si diagram (see **Fig. 1**), followed by an austempering step in a salt bath. In the intercritical austenitization stage, a partial transformation of ferrite into austenite (depending on the austenitizing temperature) takes place (**Fig. 2**).

The amount of ferrite increases as the austenitization step is closer to the lower critical temperature (Lct). On the other hand, when using high austenitizing temperatures, close to the upper critical temperature (Uct), the amount of allotriomorphic ferrite decreases and it is present as a dispersed microconstituent in an austenitic matrix. Then, during the austempering stage, the austenite transforms into ausferrite (see Fig. 2). Therefore, the final phases in the microstructure of Dual Phase ADI, obtained from this thermal cycle, are free ferrite and ausferrite. This heat treatment has allowed to obtain microstructures with different amounts and morphologies in both phases, depending on the heat treatment

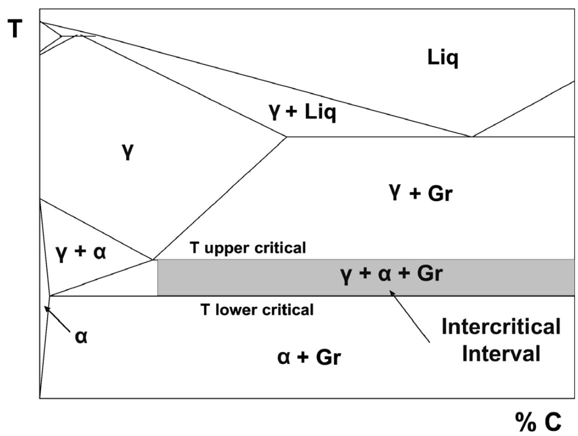

Fig. 1. Representation of Fe–C phase diagram (at 2.5%Si).[12)]

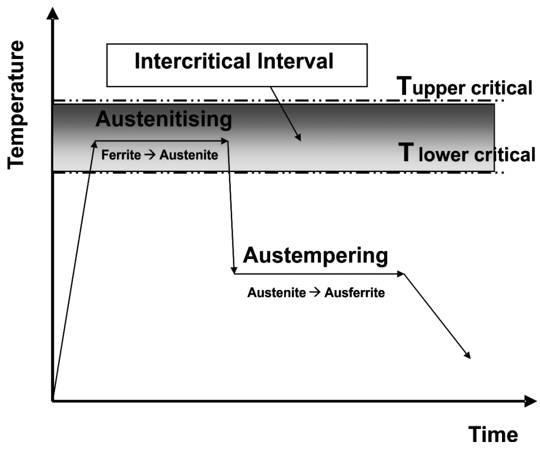

Fig. 2. Heat treatment employed to obtain Dual Phase ADI.

parameters selected.

This new type of DI has awoken technological interest and focused research efforts on determining and improving the mechanical properties of Dual Phase ADI and analyzing new parameters to optimize production methodologies.

1.1. Influence of Silicon Contents on Ductile Iron

Indeed the chemical composition of the melt has a strong influence on DI microstructure and properties. Silicon is one of the most common elements always present in DI chemical composition. This element exerts a strong effect on the impact toughness and tensile properties of ferritic DI. As the silicon content increases, the yield and tensile strength increase too. However silicon increase raises the ductile-brittle transition temperature and has a detrimental effect on the ductility of ferritic DI.[13)]

On the other hand, the literature sustains that the position and amplitude of the interval is substantially modified by the alloy chemical composition.[14,15)] In particular, silicon promotes ferrite formation in as-cast conditions and has a strong effect on the intercritical interval. Given this, the presence of this element in properly adjusted amounts could result in a fully ferritic microstructure in as-cast conditions. This could be highly beneficial for the manufacturing of mechanical components with Dual Phase ADI structures because: 1) The prior costly annealing heat treatment could be avoided, and 2) The dimensional changes undergone by the parts with Dual Phase ADI structures during the austempering thermal cycle could be minimized, if the data in the literature stating that dimensional changes are minimal when the starting microstructure is fully ferritic[16,17)] are considered.

This work explores the influence of silicon content on DI as-cast microstructures and mechanical properties, as well as its effect on the upper and lower critical temperatures. Its ultimate aim is to optimize the production process of Dual Phase ADI by reducing operating costs through the appropriate design of the chemical composition.

2. Experimental Methodologies

2.1. Melts

To analyze the influence of silicon content on the transformations occurring within the intercritical interval as well as on the mechanical properties, four DI melts with different silicon levels, ranging from 2.4 to 4.2%, were produced. The melts were obtained from a metal casting foundry, using a 500 kg capacity medium frequency induction furnace and regular quality raw materials. Inoculation and nodulization procedures were carried out by using conventional techniques. The melts were alloyed with cooper to improve austemperability, and were poured into 25 millimeter thick Y-blocks (ASTM A 395), used to prepare test specimens. The chemical composition of the melts was determined by means of a Baird spark emission optic spectrometer. The metallographic sample preparation was conducted by using standard techniques. The microstructural characterization was made by optical microscopy. Metallographic etching was performed with 2% nital. The nodularity and nodule count values were determined according to ASTM A 247 standard.

2.2. Heat Treatments

The study of the intercritical interval of each alloy involves determining the upper and lower critical temperatures, and the percentages of phases (ferrite and austenite) as a function of the holding temperature within the intercritical interval. The methodology employed to establish the intercritical interval for a specific alloy has been detailed in previous works,[4–6)] and is herein summarized as follows: several specimens of each melt 12 mm in diameter × 25 mm in length were subjected to annealing thermal cycles consisting of: a) austenitizing at 900°C for 3 hours, b) cooling down to 740°C inside the furnace, c) holding at 740°C for 10 hours, and d) cooling down to room temperature inside the furnace. To establish the intercritical interval for each melt, after annealing, the samples were subjected to thermal cycles involving austenitizing stages ranging from 730°C to 900°C at steps of 10°C. Each complete thermal cycle consisted of holding the sample for one hour in the furnace at a selected austenitizing temperature ($T\gamma$). Samples were water quenched after the austenitizing step. The resulting microstructures were composed of different amounts of ferrite (original matrix) and martensite (quenched austenite), depending of the selected austenitizing temperature.

The ferritizing treatments as well as all the austenitizing steps were carried out in electric furnaces, while a 500 kg salt bath was used to perform the austempering steps.

The micro-constituents were quantified using an optical microscope and Image Pro Plus software. Reported values are the average of at least five determinations. Graphite areas are not included in the percentages of the reported phases.

2.3. Mechanical Properties

Brinell hardness (HBW) was measured using a 2.5 mm tungsten carbide ball and a bench tester with a 187.5 kg load. The influence of the silicon level on the ductile-brittle transition temperature was analyzed for fully ferritic structures in each melt. For that purpose, impact toughness was evaluated by means of a Charpy impact test, carried out according to the ASTM E 23 standard, using 10 mm×10 mm×55 mm un-notched specimens and an AMSLER pendulum with an initial energy of 300 J (5 m/s impact speed). The reported values are the average of 4 determinations. Tensile tests were conducted in agreement with ASTM E8M/88 standard, using a universal testing machine (INSTRON 8501).

3. Results and Discussion

3.1. Metallographic Characterization in As-cast Condition

Table 1 lists the chemical compositions of the melts, identified as CQ1, CQ2, CQ3 and CQ4, respectively. The metallographic characterization in as-cast condition for all melts is detailed in **Table 2**. **Figure 3** illustrates the microstructures obtained in as-cast condition for the different melts. As it can be observed, as the silicon content increases, so does the amount of ferrite.

Table 1. Chemical compositions of the melts.

Melt	C	Si	Mn	Mg	Cu	P, S	C.E.
CQ1	3.32	2.40	0.37	0.64	0.006	< 0.017	4.1
CQ2	3.36	3.13	0.32	0.50	0.025	< 0.025	4.4
CQ3	2.81	3.54	0.36	0.64	0.011	< 0.024	4.0
CQ4	2.64	4.20	0.35	0.62	0.011	< 0.021	4.0

Table 2. Metallographic characterization in as-cast condition.

Melts	CQ1	CQ2	CQ3	CQ4
Silicon content [%]	2.4	3.1	3.5	4.2
Nodularity	80	90	80	80
Nodular size	5	5	5	5
Nodular count [nod/mm^2]	100	100	100	100
Graphite [%]	10	10	10	10
Ferrite [%]	26	39	70	80
Pearlite [%]	64	51	20	10

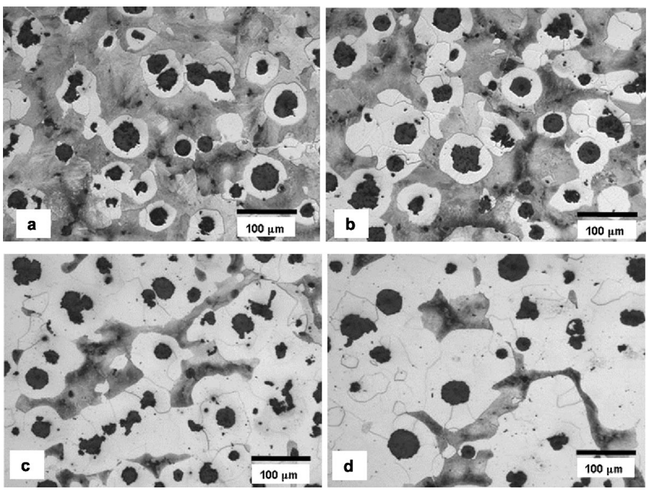

Fig. 3. Microstructures in as-cast condition for the different melts: (a) CQ1, (b) CQ2, (c) CQ3, (d) CQ4.

3.2. Metallographic Characterization of the Intercritical Interval for the Different Melts

The annealing heat treatment allowed to obtain a fully ferritic microstructure for melts CQ2, CQ3, and CQ4, while melt CQ1 (with the lowest Si content) showed globulized cementite located in the last to freeze zones ("LTF" zones). This type of structure is depicted in **Fig. 4**.

As already defined by the authors in previous works,[4–6] the lower critical temperature (Lct) is the lower temperature at which the austenite transformation starts (detected by the presence of less than 5% of martensite after quenching). The upper critical temperature (Uct), in turn, is defined as the temperature at which a matrix with over 98% of martensite is detected after quenching the samples held at such temperature. The critical temperature values as a function of the silicon content of the melt are shown in **Fig. 5**.

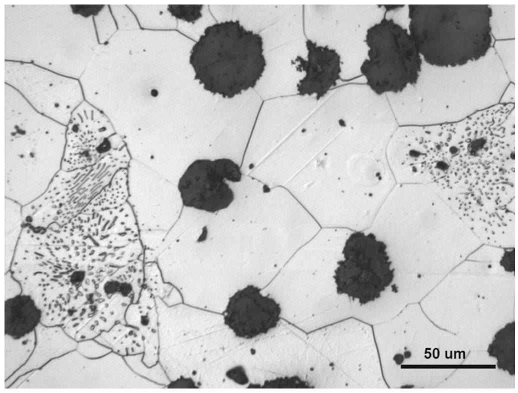

Fig. 4. Microstructure of melt CQ1 after annealing heat treatment.

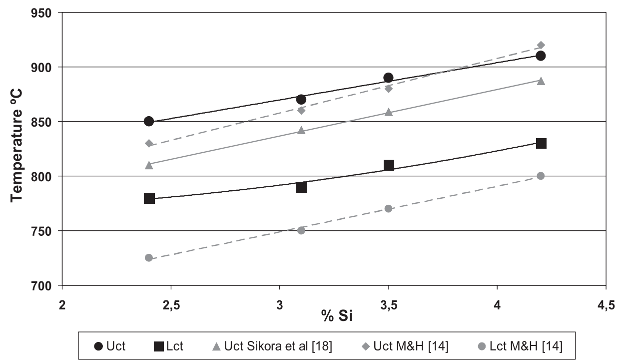

Fig. 5. Critical temperature values as a function of silicon content in each melt.

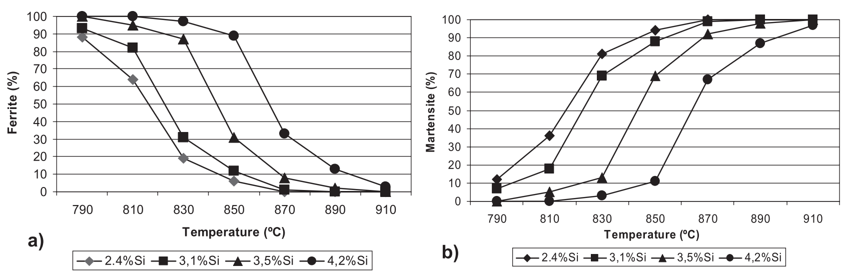

Fig. 6. Amount (in %) of ferrite (a) and martensite (b) as a function of the intercritical austenitizing temperature of each melt.

Indeed the increase in silicon content increments the upper and lower critical temperatures, while the amplitude of the intercritical interval remains nearly constant for the melts. The results predicted by Sikora *et al.*[18] and Maitland & Hughes[14] as a function of silicon content are also shown in Fig. 5. It can be concluded that the results obtained in this work are in reasonable agreement with the values reported in the literature. **Figures 6**(a) and 6(b) illustrate the amount (in percentage) of ferrite and martensite as a function of the intercritical austenitizing temperature of each melt.

3.3. Mechanical Properties

3.3.1. Hardness and Tensile Properties

Table 3 lists the average values of hardness and tensile properties for different as-cast and annealed structures, depending on the silicon level in the melt. For the as-cast samples, the values obtained indicate that when the amount of silicon is increased, tensile strength, yield stress and hardness decrease, while elongation until failure increases. Higher silicon levels promote an increase in ferrite in the microstructure and reduce the amount of pearlite (see Table 2). Conversely, regarding fully ferritic samples, as the silicon level increases, hardness, tensile strength and yield stress increase as well, while elongation until failure decreases. The Si content exerts a strong influence on the mechanical properties of the ferrite phase. This element forms a substitutional solid solution with Fe, resulting in an increase in the hardness, tensile strength and yield stress of ferrite but decreasing its ductility (ferrite embrittlement).[13] However, melt CQ 1 (2.4% Si) yielded the lowest elongation value until failure, attributed to the presence of globulized cementite.

3.3.2. Impact Properties

To evaluate the influence of silicon content on ductile-brittle transition temperature, Charpy tests were conducted on fully ferritic structures for each melt. **Figure 7** shows the average values of impact toughness as a function of the test temperature for the different melts. As it can be clearly noticed, when silicon content increases, the ductile-brittle transition temperature rises. These results are consistent with the values reported in the bibliography.[13] It is worth pointing out that, at room temperature, melt CQ4 exhibits brittle behavior; whereas melt CQ2 (Si%=3.1) yields highest values in the upper shelf zone. This distinctive difference deserves further analysis.

Table 3. Values of tensile properties for different as-cast and after annealing heat treatment structures, depending on the silicon level in the melt.

Condition	As-cast				After annealing			
Melt	CQ1	CQ2	CQ3	CQ4	CQ1	CQ2	CQ3	CQ4
σ_{max} [MPa]	725	688	622	612	401	459	482	542
$\sigma_{0.2}$ [MPa]	476	460	465	453	281	307	320	410
δ[%]	9	11	12	13	19	21	15	13
Hardness [Brinell]	251	215	208	204	151	160	175	197

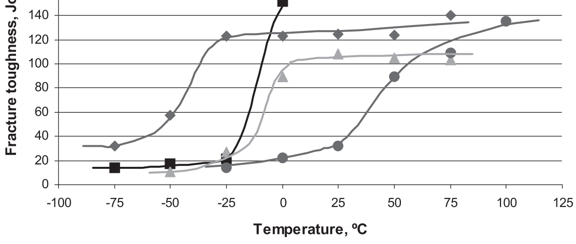

Fig. 7. Influence of silicon level on impact toughness and ductile-brittle transition temperature.

3.4. High Silicon Dual Phase ADI Microstructures

As it was mentioned above, Dual Phase ADI microstructures are composed of different amounts of allotriomorphic ferrite and ausferrite. Given the fact that this work intends to analyze the influence of silicon content on this new kind of microstructures, several intercritical thermal cycles, such as that shown in Fig. 2, were performed to obtain microstructures with different amounts of ferrite and ausferrite. **Figure 8** depicts the micrographs corresponding to samples submitted to different intercritical austenitizing temperatures and austempered at 350°C for all the melts studied. This figure depicts Dual Phase ADI microstructures with approximately 50% of ferrite and 50% of ausferrite. As it can be seen, austenite nucleation and growth develops mainly at LTF zones and at ferritic grain boundaries.

It is worth underlining that one of the objectives of the present work was to study the feasibility of obtaining Dual Phase ADI structures using high silicon DI melts. The results obtained in this work suggest that high silicon ductile iron (Si content over 3.0%) can be used in the manufacturing of cast parts with "ADI Dual Phase" microstructures, with prospective advantages in the production process, in comparison to lower silicon content ductile irons.

As it was mentioned in the Introduction, the use of high silicon DI would be advantageous for manufacturing components with Dual Phase ADI structures, since it does not require prior annealing heat treatments, could minimize dimensional changes and increases machinability in the as-cast conditions. **Figures 9** and **10** illustrate Dual Phase ADI structures obtained under as-cast conditions and after

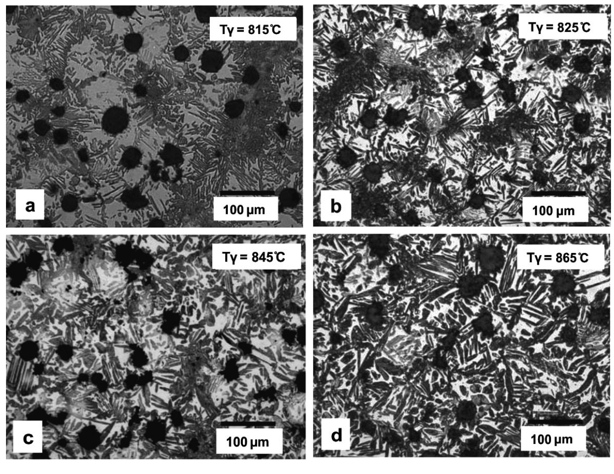

Fig. 8. Micrographs corresponding to Dual Phase ADI microstructures for melts CQ1, CQ2 CQ3 and CQ4, respectively (T austempering = 350°C).

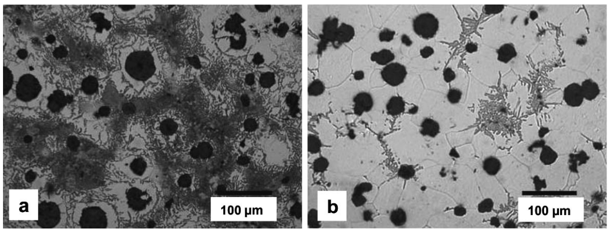

Fig. 9. Micrographs corresponding to Dual Phase ADI microstructures for melts CQ1 obtained from: a) as-cast condition and b) fully ferritic matrix. Austenitizing at 795°C for 2 hours and austempering at 350°C for 90 minutes.

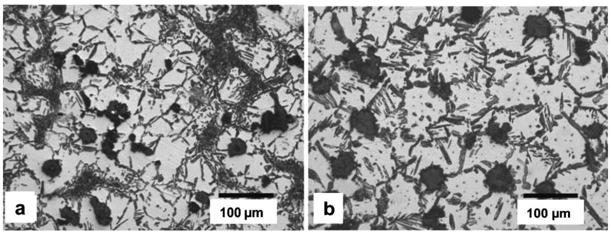

Fig. 10. Micrographs corresponding to Dual Phase ADI microstructures for melts CQ3 obtained from: a) as-cast condition and b) fully ferritic matrix. Austenitizing at 825°C for 2 hours and austempering at 350°C for 90 minutes.

annealing heat treatment (fully ferritic matrix) for CQ1 (Si%=2.4) and CQ3 (Si%=3.6) melts, respectively. The samples were austenitized at different temperatures for 2 hours and austempered at 350°C for 90 minutes. It can be observed that after 2 hours of the intercritical austenitizing step, the as-cast sample corresponding to CQ1 melt (Fig. 9(a)) does not reach the equilibrium phase percentage, while it is achieved by the Dual Phase ADI sample obtained from the fully ferritic matrix (Fig. 9(b)).

However, regarding CQ3 melt, which features higher silicon content and higher ferrite percentage in the microstructure under as-cast conditions, the equilibrium phase percentage is achieved after 2 hours of maintaining it at the intercritical austenitizing temperature (see Fig. 10). These results indicate that high silicon DI (with content higher than 3%) would yield Dual Phase ADI microstructures able to dispense with prior annealing heat treatments.

Larker *et al*. explored some other features of high silicon ferritic DI with a view to improving the technical and economical advantages of DI and to promoting the supply of heavy cast parts of greater material grade.[19]

Still the main goal of this line of research is to optimize production technologies and mechanical properties of Dual Phase ADI. To complete this ultimate objective, new research that promises to advance our understanding of mechanical properties is currently under way. In particular, tensile properties and impact toughness of high silicon Dual Phase ADI structures are being studied.

4. Conclusions

(1) The influence of Si content on the upper and lower critical temperatures, intercritical interval amplitude, microstructural characteristics and mechanical properties of ductile irons with Si content ranging from 2.4 to 4.2% was studied. The results revealed that as silicon content increases, so do the upper and lower critical temperatures, while the amplitude of the intercritical interval remains nearly constant for all melts.

(2) The mechanical characterization was performed on as-cast and fully ferritic microstructures as a function of the melt silicon content. For as-cast samples, the values obtained indicate that by increasing the amount of silicon, tensile strength, yield stress and hardness decrease, while elongation until failure increases. This is explained by the fact that the higher silicon levels in the melt increase the amount of ferrite in the microstructure and reduce the amount of pearlite.

(3) The mechanical properties of the ferritic phase show a strong influence with the percentage of Si. In fully ferritic DI samples, as the silicon level increases so does hardness, tensile strength and yield stress, while elongation until failure decreases.

(4) Impact properties revealed that when silicon content increases, the ductile-brittle transition temperature rises. The melt with the highest silicon level (4.2%) yielded brittle behavior at room temperature.

(5) The results obtained in this work suggest that high silicon ductile iron (Si content around 3.0%) could be used in the manufacture of cast parts with "Dual Phase ADI" microstructures, with prospective advantages in production process and performance, in comparison with DI with lower silicon content. The use of high silicon DI obviates the need of prior annealing heat treatments, could minimize dimensional changes of Dual Phase ADI parts, and increases the machinability of pieces in as-cast condition.

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