

Review

Slightly Irregular Spheroidal Graphite (Type-V, ISO): Typical Graphite Morphology for High-Si/SiMo Ductile Cast Irons

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Abstract

The present paper reviews original data obtained by the authors from recent separate publications with additional unpublished data, specifically concerning the specific graphite morphology in Si/SiMo alloyed ductile irons (2.5–5.5% Si, 0.01–2.3% Mo) with alloying grade, cooling rate, and inoculation as influencing factors. Different formulas used for nodularity evaluation, including different forms of graphite participating in different proportion and two graphite shape factors: Roundness (RSF, involving maximum Ferret) and Sphericity (SSF, involving real perimeter). Slightly irregular spheroidal graphite morphology (Form V, ISO 945) characterized by $RSF = 0.59–0.75$ was found to be typical for Si and SiMo ductile irons in all of the test conditions, such as Si level (4.2–5.25% Si), Si–Mo system (4.1–4.8% Si + 1.6–2.3% Mo), mould type (green sand, resin sand, metal mould, external metallic chill), un-inoculation and inoculation, and inoculating element type. Increasing Si content negatively affects the compactness degree of spheroidal graphite particles (transition from VI to V form) and nodularity. Metal mould versus sand mould solidification of 4.5% Si ductile iron increases nodule count, graphite shape factors ($RSF = 0.68–0.7$ versus $0.59–0.64$) and nodularity (67% to 76%). An external metallic chill in a resin sand mould promoted directional solidification and showed a nodularity decrease for increasing Si, for all the nodularity formulas and for the cooling rate range. Inoculating elements influenced the shape factors in thin wall castings, where Ca–Ba was better than simple Ca and Ca–RE could promote graphite at higher real perimeter and with lower shape factors. The correlation of the aspect of structure, graphite parameters and nodularity led to the conclusion that the nodularity formula according to ISO/WD 945-4-2015 (100% total area of Form VI and 90% of Form V), with SSF instead of RSF, appears to be better in High-Si/SiMo DI (especially for more than 4% Si), because it takes into account the presence of slightly irregular spheroidal graphite (with high real perimeter) at a high rate.

Keywords: ductile cast iron; Si and Si–Mo alloying; inoculation; solidification cooling rate; spheroidal/nodular graphite; metal mould; sand mould; metallic chill; graphite particle perimeter; graphite shape factors; graphite nodularity



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1. Introduction

Silicon is an essential element in ductile (spheroidal/nodular graphite) cast irons, defining these materials in the ternary Fe–C–Si system. Commercial cast irons also contain other elements (more than 30, such as X = Mn, P, S, Al, Cr, Ni, Cu, etc., depending on the charge materials and melting/treatment procedures). Figure 1 shows the major groups of commercial cast irons according to Si content, within the general range of 1.8–18 wt.% Si:

- (A) Conventional ductile irons (1.8–3.0% Si), with a ferrite/ferrite–pearlite/pearlite metal matrix, for general applications (higher pearlite/ferrite ratio, higher strength and hardness and lower ductility and toughness characteristics); [ASTM A 536-84 [1]; 2019; EN 1563:2018 [2]; ISO 1083:2018 [3]; (a) ferritic to pearlitic spheroidal graphite irons section].
- (B) Solution-Strengthened High-Si Ferritic Ductile Cast Iron (SSFDI) [EN 1563:2018 [2]; ISO 1083:2018 [3]; (b) solid-solution strengthened ferritic spheroidal graphite cast irons section]; a more predictable and controlled ferrite replaced the unstable mixed ferrite–pearlite matrix, at reduced hardness variation and predictable machinability ($R_m = 450\text{--}650\text{ MPa}$; $R_{p0.2} = 350\text{--}500\text{ MPa}$; $A = 10\text{--}20\%$), usually used in automotive industry. Si-alloying promotes ferrite at higher strength and hardness characteristics, but at lower ductility and toughness.
- (C) Si (4–6% Si) and Si–Mo (2.5–5.5% Si + 0.2–2% Mo) ductile cast irons for resistance to oxidation and corrosion at elevated temperatures [SAE J2582 (2004) [4]]. Mo addition favours superior mechanical properties, especially at elevated temperatures ($R_m = 400\text{--}650\text{ MPa}$; $R_{p0.2} = 250\text{--}550\text{ MPa}$; $A = 3\text{--}12\%$), typically for exhaust applications.
- (D) Cast irons for higher resistance to corrosion, with 14–18% Si and a ferritic matrix; [Corrosion resistant High-Si iron castings, lamellar graphite, ASTM A518/A518M-99 (2018) [5]].

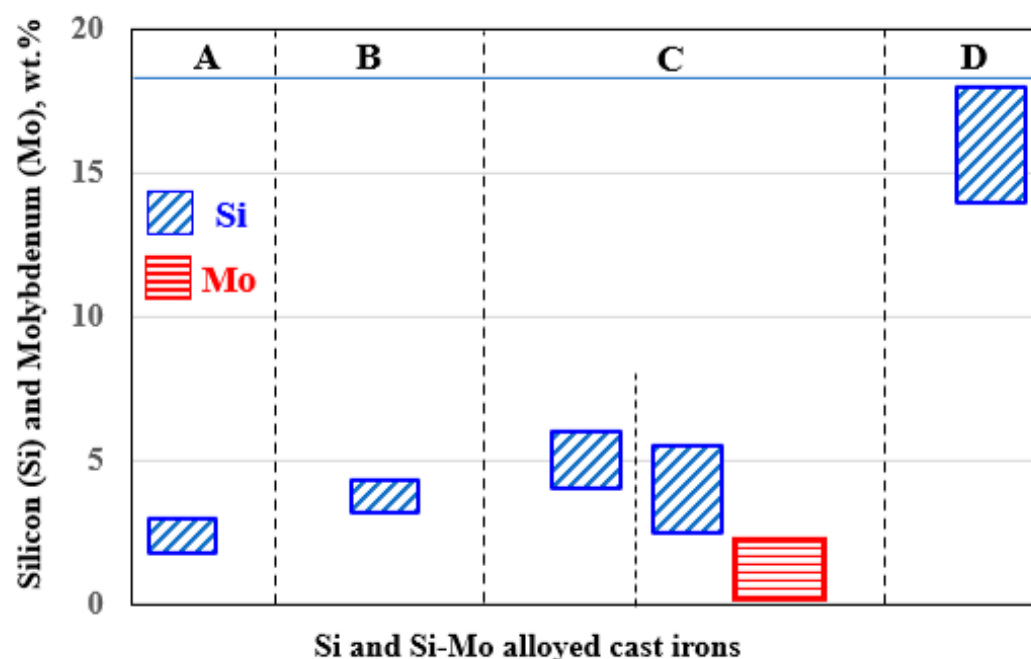


Figure 1. Si and SiMo alloyed cast irons: (A) Conventional ductile irons, ferritic to pearlitic spheroidal graphite irons; (B) Si-solid-solution strengthened ferritic spheroidal graphite irons; (C) Si and (Si-Mo) alloyed spheroidal graphite irons for high temperatures, oxidation and corrosion resistant; and (D) high-corrosion resistant high-Si grey iron castings.

Silicon affects both the solidification (promoting graphite instead of carbides) and the solid state (eutectoid) transformation (promoting ferrite instead of pearlite and ferrite alloying), independently of the graphite morphology.

Important research programs have pointed out the detrimental effects of silicon alloying on the spheroidal morphology of graphite. A high incidence of chunky graphite was found, generally negatively affecting the mechanical properties [6–8], such as for 4.5% Si compared to 3.5% Si [5] in the castings body with a slow cooling rate (Ni promotes chunky graphite only in the thermal centre) [7]; however, not in all cases was Si found to be a

promoter of chunky graphite [9]. The higher the silicon content and lower the solidification cooling rate, the higher the risk of chunky graphite formation [6–8,10]. Vanadium promoted chunky graphite in 3.8% Si ductile iron casting with a long solidification time [10]. To counteract the chunky graphite formation because of silicon alloying, Ce/Sb addition could usually be a solution [11]. Bi-containing inoculants are effective at improving the graphite morphology in high-Si ductile iron castings [12,13]. The addition of 0.01% Bi gave the best results, where the Ce/Bi ratio amounted to 0.6 [14]. Controlled Bi/Ce addition (3.2% Si–3.2% C–75 mm-thick Y–IV keel block casting) counteracted the contamination of iron, though a VI-graphite form will not be achieved [15].

Various molten-iron treatment techniques improve the quality of high-Si ductile iron production. For example, thin-wall castings can benefit from a two-step liquid treatment using a RE-bearing FeSiCaMg master alloy followed by a Ca, Ba–FeSi inoculant [16]. An effective treatment package, combining Ba preconditioning with Bi/RE-bearing inoculation, is recommended for foundry production of this material [17]. Optimized properties require well-shaped nodules, obtained through special inoculation techniques adapted to the high Si content and solidification rate; otherwise, graphite degeneration may occur [18]. Different production solutions for solution-strengthened ferritic ductile irons obtained by varying the MgFeSi alloy, cover material, and inoculating materials [19]. In addition, inoculant type influenced the porosity sensitivity of high-Si ductile irons [20].

High Si (3–6% Si) and Si–Mo (2.5–5.5% Si + 0.2–2% Mo) ductile cast irons have been receiving a lot of attention lately due to their properties and areas of use. Recent research works pointed out not only the attractive properties of these materials but also possible negative effects of Si-alloying on the spheroidal graphite phase quality, mainly as chunky graphite morphology occurrence and/or lower compactness degree of obtained spheroidal particles [6–8,10–19]. This information is still disparate, without a unitary approach.

The research programs of the present authors focused on the graphite characterization in high Si/SiMo ductile cast irons obtained under different conditions, such as metallurgical treatments and solidification. The present paper reviews original data obtained by the authors from recent separate publications, referring to five research programs, with additional unpublished data, specifically concerning the specific graphite morphology in Si/SiMo alloyed ductile irons (2.5–5.5% Si, 0.01–2.3% Mo). Alloying grade, solidification cooling rate (casting wall thickness; metal mould-MM vs. sand mould-SM; solidification in the metallic chill) and the inoculation/inoculating elements (Ca, Ba, RE) were considered as influencing factors. For each presented research program, the already representative published information is summarized, with the addition of new data referring to graphite specific size and compactness parameters and the graphite nodularity involving them. Special attention was paid to graphite nodularity, which is an important condition for the final acceptance of the castings. In this way, an overview of the specific graphite phase in high Si/SiMo ductile cast irons is offered, with further documentation than the results of the separate publications.

2. Materials and Methods

Tables 1 and 2 summarize the main parameters of the ductile cast irons, evaluated in five research programs, in terms of their major objectives and experimental procedure parameters (Table 1) and the final chemical compositions obtained (Table 2). More data was obtained from the results previously reported, such as Program I [21,22], Program II [23], Program III [24,25], Program IV [26] and Program V [16,25].

Table 1. Experimental parameters.

Pr	Objective	Iron	Melting	Mg-Treatment	Inoculation	Mould-Samples	Ref.
I	Effect of Si/SiMo (4% Si vs. 2.5% Si)	1, 2, 3	10 kg, CIF, 8000 Hz	Tundish Cover 2.0 wt.% FeSiCaMgRE	Pouring Ladle 0.3 wt.% Ca, Ce, S, O-FeSi	RSM 20 mm dia bars	[21,22]
II	Graphite nodularity evaluation vs. shape factors	4	10 kg, CIF, 8000 Hz	Tundish Cover 1.5 wt.% FeSiCaMgRE	No-inoculation	GSM Wedge samples	[23]
III	Metal Mould (MM) vs. Sand Mould (SM)	4	10 kg, CIF, 8000 Hz	Tundish Cover, 1.5 wt.% FeSiCaMgRE	No-inoculation	GSM MM Wedge samples	[24,25]
IV	Metallic Chill solidification	4, 5, 6	10 kg, CIF, 8000 Hz	Tundish Cover 1.5 wt.% FeSiCaMgRE	No-inoculation	RSM, Metallic chill Samples 6.5 × 56 × 60 mm	[26]
V	Ca/Ca,Ba/Ca,RE inoculation	7	10 kg, CIF, 8000 Hz	Tundish Cover 1.5 wt.% FeSiCaMgRE	In-Mould, 0.1 wt.% Ca-FeSi Ca,Ba-FeSi Ca,RE-FeSi	RSM 20 mm dia bars	[16,25]

CIF—coreless induction furnace; RE—rare earth; RSM—resin sand mould; GSM—green sand mould; MM—metal mould; and Ref.—reference.

Table 2. Chemical composition of the test ductile cast irons (wt.%).

Iron	C	Si	Mn	Mo	Mg	Cr	Ni	Cu	Al	Ti	CE *	Pr
1	3.20	2.55	0.38	0.012	0.032	0.05	0.04	0.06	0.014	0.013	4.03	I
2	3.08	4.20	0.45	0.011	0.057	0.05	0.04	0.06	0.019	0.014	4.42	I
3	2.76	4.10	0.34	1.66	0.036	0.05	0.04	0.06	0.021	0.013	4.06	I
4	3.33	4.55	0.22	0.07	0.035	0.05	0.10	0.10	0.006	0.006	4.71	II, III, IV
5	3.46	5.25	0.22	0.04	0.045	0.09	0.06	0.11	0.007	0.007	5.05	IV
6	3.30	4.80	0.22	2.31	0.045	0.06	0.22	0.08	0.002	0.007	4.75	IV
7	3.27–3.35	4.34–4.58	0.20–0.25	0.065–0.080	0.032–0.036	0.05–0.06	0.10–0.24	0.17–0.21	0.006		4.64–4.68	V

* CE—carbon equivalent, CE = % C + 0.3 (% Si + % P)—0.03% Mn + 0.4% S; and Pr—program.

A reference conventional cast iron (Iron 1, Group A-Figure 1) was used, at 2.55% Si, 3.20% C, 0.38% Mn, 0.032% Mg_{res} and 4.03% Carbon Equivalent. High-Si (4.2–5.25% Si, Irons 2, 4, 5, 7) and high-SiMo (4.1–4.8% Si + 1.6–2.3% Mo, Irons 3, 6), corresponding to Group C (Figure 1) solidification under different conditions, as concerns the variation of the solidification cooling rate and inoculation application. All the tested cast irons were obtained in closed melting and Mg-treatment conditions.

For high precision metal analysis, a SPECTROLAB high-end spectrometer (SPECTRO Analytical Instruments GmbH, Kleve, Germany) was used, with simultaneous detection by hybrid optics (photomultiplier tubes) and the Spectroscopic Charge Coupled Device detection system. The instrument achieves detection limits below 1 mg/kg.

The graphite characteristics were evaluated using Automatic Image Analysis [analySIS FIVE Digital Imaging Solutions software (Version 5.0)] and Scanning Electron Microscopy. Graphite particles at minimum 5 µm size were considered (according to the stipulation in the software).

The present paper focuses on the graphite characterization in Si/SiMo alloyed ductile cast irons, as regards the graphite particles parameters and graphite nodularity. Graphite particles are characterized by the minimum ($F_{\min}$) and maximum Ferret ($F_{\max}$), area (A_G), convex (P_c) and real (P_r) perimeter, according to Figure 2, which also defines different graphite particle shape factors, such as the Aspect Ratio (AR), Elongation (E) and Convexity (Cv).

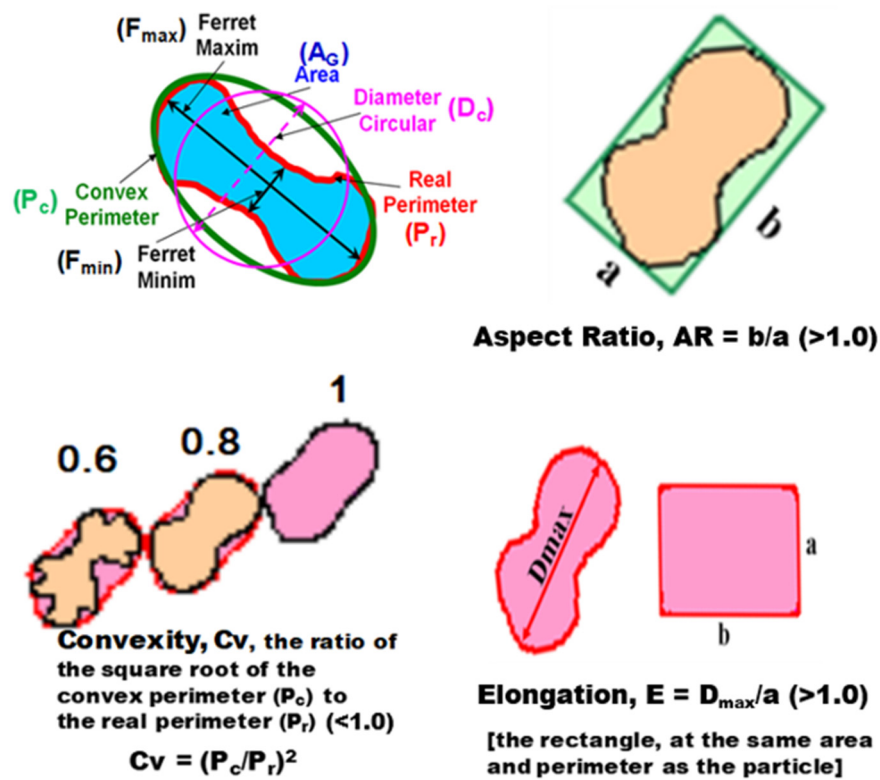


Figure 2. Graphite particle parameters considered for shape factors and nodularity [23].

More complex shape factors considered were the Roundness Shape Factor (RSF, Equation (1) and Figure 3) and the Sphericity Shape Factor (SSF, Equation (2)). Both involve the area of the graphite particle (A_G), differentiated by the second parameter, such as F_{max} for RSF and P_r for SSF, respectively.

$$RSF = 4A_G / \pi F_{max}^2 \tag{1}$$

$$SSF = 4\pi A_G / P_r^2 \tag{2}$$

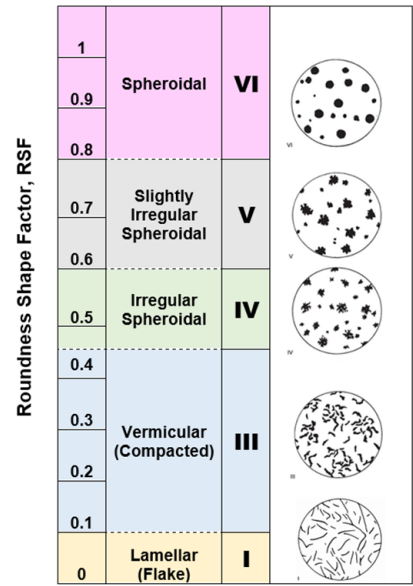


Figure 3. Representative graphite forms (ISO 945) characterized by Roundness Shape Factor (RSF).

3. Results

3.1. Graphite Characterization—Shape Factors and Nodularity

According to the CGI-ISO 16112:2017 Standard [27] (usually used in compacted/vermicular graphite iron castings production), the graphite particles are defined as nodules ($RSF = 0.625–1.0$), intermediates ($RSF = 0.525–0.625$), and vermicular/compacted ($RSF < 0.525$).

ISO/WD 945-4:2015 [28], finalized by ISO-945-4:2019 (2024) [29] and applied to ductile cast irons, refer to the spheroidal graphite ($RSF = 0.45–1.0$), vermicular/compacted graphite ($RSF = 0.10–0.45$), and lamellar graphite ($RSF < 0.10$), with three classes of spheroidal graphite, namely the spheroidal ($RSF \geq 0.80$), slightly irregular spheroidal ($RSF = 0.60–0.80$) and irregular spheroidal ($RSF = 0.45–0.60$), according to Figure 3, obtained on the base of references [28,30].

Graphite nodularity (NG) is a delicate parameter of ductile (spheroidal graphite) cast iron production and its quality evaluation. Nodularity is a condition of acceptance of ductile iron castings, usually regulated at a minimum 80% level (70% or 90% in any cases). Nodularity expresses the share of spheroidal (nodular) graphite in the total graphite phase. The results obtained are influenced by a larger number of factors, depending on the analysis technique, considered graphite parameters (the graphite particles area or number, the minimum size), the definition/acceptance of the spheroidal morphology, etc.

The progress in automatic image analysis has led to a more accurate assessment of the graphite nodularity in cast irons, based on the considered complex shape factors, including the representative parameters of the graphite particles.

The Roundness Shape Factor (RSF , Equation (1)) is usually used to evaluate the nodularity for compacted graphite cast iron (CGI, ISO 16112:2017 [27]), (NG1–Equation (3)) and ductile (spheroidal graphite) cast irons [DI, ISO/WD 945-4:2015 [28] and ISO 945-4:2019 [29]], but at different ranges of RSF , which is involved at different values.

The nodularity NG2 formula (Equation (4)) was introduced by ISO/WD 945-4:2015 (Working Draft) [28], considering only the last two forms of spheroidal graphite (V—slightly irregular spheroidal and VI—spheroidal, Figure 3) and including 100% total area of Form VI and only 90% area of Form V. This NG2 formula was used from the beginning of the actual working program referring to high-Si ductile cast irons [21,22].

Later, the nodularity formula was introduced [P_{nod} (NG), Equation (5), ISO-945-4:2019 (2024) [29]], which also kept the consideration of only the V and VI forms of graphite but changed their participation in the nodularity formula, as 100% for each one.

As it was found that high-Si ductile irons are sensitive to V-graphite morphology formation, resulting in a structure with V form graphite at high rate [16,21–26], the graphite nodularity formula NG2 (Equation (4), ISO/WD-945-4:2015) [28] was kept, despite the fact that the actual ISO-945-4:2019 (2024) [29] includes a changed nodularity formula.

Nodularity NG3 (Equation (6)) resulted from Equation (4), where Roundness Shape Factor (RSF) replaced by Sphericity Shape Factor (SSF).

Nodularity NG4 (Equation (7)) and NG5 (Equation (8)) express the rate of the graphite particles at a minimum considered shape factor (RSF or SSF) in the total graphite phase.

$$NG1 = [(\Sigma A_{\text{particles}}(RSF=0.625–1.0) + 0.5 \cdot \Sigma A_{\text{particles}}(RSF=0.525–0.625)) / \Sigma A_{\text{tot}}] \times 100[\%] \quad (3)$$

$$NG2 = [(\Sigma A_{\text{particles}}(RSF \geq 0.8) + 0.9 \cdot \Sigma A_{\text{particles}}(RSF=0.6–0.8)) / \Sigma A_{\text{tot}}] \times 100[\%] \quad (4)$$

$$P_{nod} [NG] = [(A_{VI} + A_V) / A_{all}] \times 100 = [\Sigma A_{\text{particles}}[RSF=0.6–1.0] / \Sigma A_{\text{tot}}] \times 100[\%] \quad (5)$$

$$NG3 = [(\Sigma A_{\text{particles}}(SSF \geq 0.8) + 0.9 \cdot \Sigma A_{\text{particles}}(SSF=0.6–0.8)) / \Sigma A_{\text{tot}}] \times 100[\%] \quad (6)$$

$$NG4 = [\Sigma A_{\text{particles}}_{\text{[RSF]}} / \Sigma A_{\text{tot}}] \times 100[\%] \quad (7)$$

$$NG5 = [\Sigma A_{\text{particles}}_{\text{[SSF]}} / \Sigma A_{\text{tot}}] \times 100[\%] \quad (8)$$

3.2. Si-Influence on the Graphite Nodularity

As result of the increasing of the Si-content in ductile cast iron, from the conventional level of 2.5% Si (Group A, Figure 1) up to a 4% Si level, typical for high-Si ductile irons (Groups B and C; Figure 1) (Program I experiments, Tables 1 and 2), important effects were obtained, as solidification pattern and structure parameters, respectively.

In a previously published paper [21], it was reported that the standard thermal analysis, focused on the representative events during solidification, identified important differences in the solidification pattern of conventional [2.5% Si], high Si [4% Si] and high Si-Mo [4% Si and 1.6% Mo, lower Si/Mo ratio = 2.5] ductile cast irons. Increasing Si-content increased the eutectic interval and decreased the undercooling during the eutectic reaction, and up to the end of solidification, in both un-inoculated and inoculated ductile cast irons. Mo additions at higher levels in high Si ductile iron decreased the eutectic interval and led to increased undercooling over the entire solidification period in un-inoculated irons. Inoculation of Si-Mo ductile irons compensates for the negative effect of Mo mainly during the eutectic reaction. Inoculation appears to be important not only in conventional ductile iron but also in high-Si irons, with the greatest effect on Si-Mo ductile irons, in controlling undercooling during solidification and of the area of porosity, including chunky graphite in the thermal centre of castings [21].

It was also found [21,22] that the deviation of the graphite particles from a reference sphere is noticeably increased by Si-alloying, a larger perimeter becoming a characteristic of the graphite particles. With more than 4% Si content, ductile irons are characterized by a medium-quality graphite phase, with prevalent Form V (ISO 945-1:2019) [31] graphite formation, including cast irons that have been inoculated. It was found that the calculated graphite nodularity increased when the minimum size of the analyzed particles decreased from 15 to 5 μm (up to 2% difference), with 10 μm as the intermediate level [22].

The sphericity (circular) shape factor [SSF, Equation (2)], which considers the real perimeter of a graphite particle was recommended in Si-alloyed ductile irons instead of the roundness shape factor [RSF, Equation (1)], involving maximum Ferret of an analyzed particle, presently incorporated in the ISO 945-4:2019 [29] standard [21,22].

More data obtained regarding the negative effect of Si/SiMo alloying on the graphite nodularity involved different calculus formulas in, such as Figure 4 shows.

By applying the graphite nodularity calculus stipulated by the ISO 16112:2017-CGI [27] (involving RSF and maximum Ferret, NG1-Equation (3)), it resulted in NG1 = 95% for 2.5% Si iron, and 79–80% for Si-alloyed irons. If the ISO/WD 945-4-2015 [28] stipulations were used, applied for ductile irons, and RSF was replaced by SSF (sphericity shape factor) (considering the real perimeter of the graphite particles), a lower level of nodularity resulted (NG3 = 72–76%), but with the same decreasing tendency as regards Si-alloying (72–74 vs. 76%).

If calculated nodularity considered a minimum level for the graphite shape factor, such as SSF (from min. 0.50 up to min. 0.80), the result is NG5 nodularity (Equation (8)). For a general acceptance of spheroidal graphite morphology (Forms IV + V + VI), more than 80% nodularity characterizes all the irons, independently of the Si-content. The higher the imposed SSF, the lower the nodularity and higher the negative effect of Si (the biggest effect on the occurrence of regular spheroidal graphite (Form VI)).

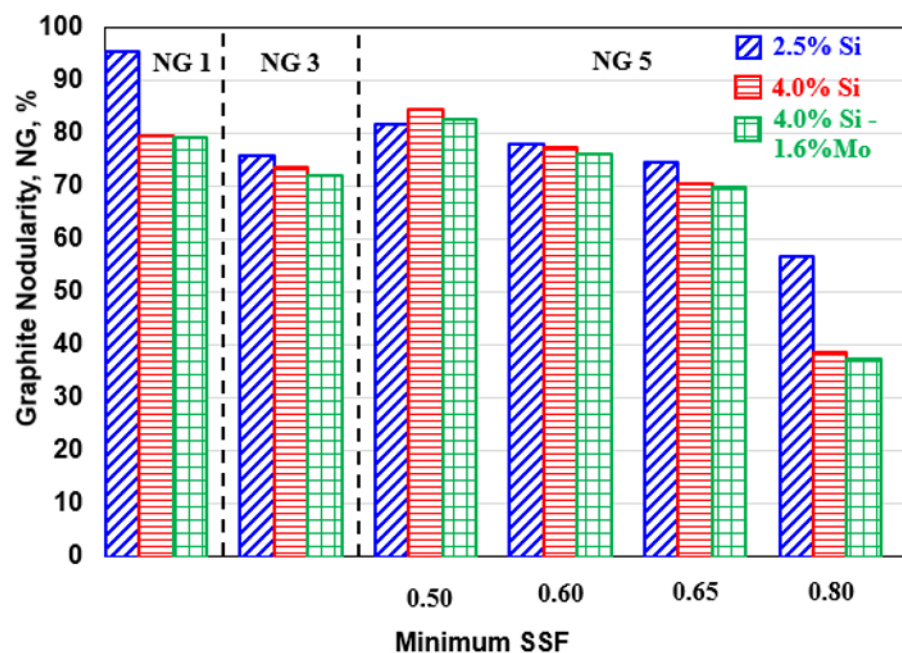


Figure 4. The influence of the alloying grade on nodularity, involving different shapes factors (Program I).

3.3. Graphite-Nodularity-Tested Calculation Formulas

An extensive research program [23] evaluated different calculus formulas for graphite nodularity evaluation in 4.5% Si, un-inoculated ductile cast iron, solidified in wedge castings in green sand mould (Program II, Tables 1 and 2). AR, E, Cv (see Figure 2), RSF (Equation (1)) and SSF (Equation (2)) as graphite shape factors, and graphite nodularity NG1, NG2 and NG3 (Equations (3), (4) and (6)) were used.

Figure 5a summarizes the obtained values [23]. The Roundness Shape Factor (RSF = 0.65–0.68) illustrates the presence of slightly irregular spheroidal graphite (Form V ISO 945-4:2019) [29], characterized by a higher real perimeter and positioned at the lower part of this field (RSF = 0.60–0.80 according to ISO 945-4-2019-DI) [29]. Other graphite shape factors, for the entire range of wall thickness of wedge castings (3–15 mm), also sustain the lower quality of this spheroidal graphite (AR = 1.32–1.39, E = 1.37–1.48, Cv = 0.91–0.92, SSF = 0.79–0.82). It was concluded that for high-Si DI (mainly >4% Si), the Graphite Shape Factor (SSF), involving the graphite particle area (A_G) and the real perimeter (P_r), is more representative than the RSF Shape Factor, based on the graphite particle area (A_G) and maximum Ferret (F_{max}). It was recommended to use a graphite nodularity calculus considering the SSF instead of the RSF, and the formula stipulated by ISO/WD 945-4-2015 [28] (NG3, Equation (6)) [18].

New data were obtained for graphite nodularity and compared to the current stipulations of the ISO 945-4-2019 [29] standard: compacted graphite cast irons standard ISO 16112:2017 [27] [NG1, Equation (3), 100% spheroidal graphite (RSF = 0.625–1.0) and 50% intermediate graphite (RSF = 0.525–0.625)]; initial introduced standard for graphite nodularity evaluation in ductile iron ISO/WD 945-4-2015 [28] [NG2, Equation (4), 100% spheroidal graphite (Form VI, RSF = 0.8–1.0) and 90% slightly irregular spheroidal (Form V, RSF = 0.6–0.8)]; and final (actual) standard form for ductile iron nodularity, ISO 945-4-2019 [29] [NG, Equation (5), 100% graphite particles V and VI forms, RSF = 0.6–1.0]. Replacing Roundness Shape Factor with Sphericity Shape Factor in the nodularity formula stipulated by ISO/WD 945-4-2015 [28] resulted in NG3 Nodularity (Equation (6)).

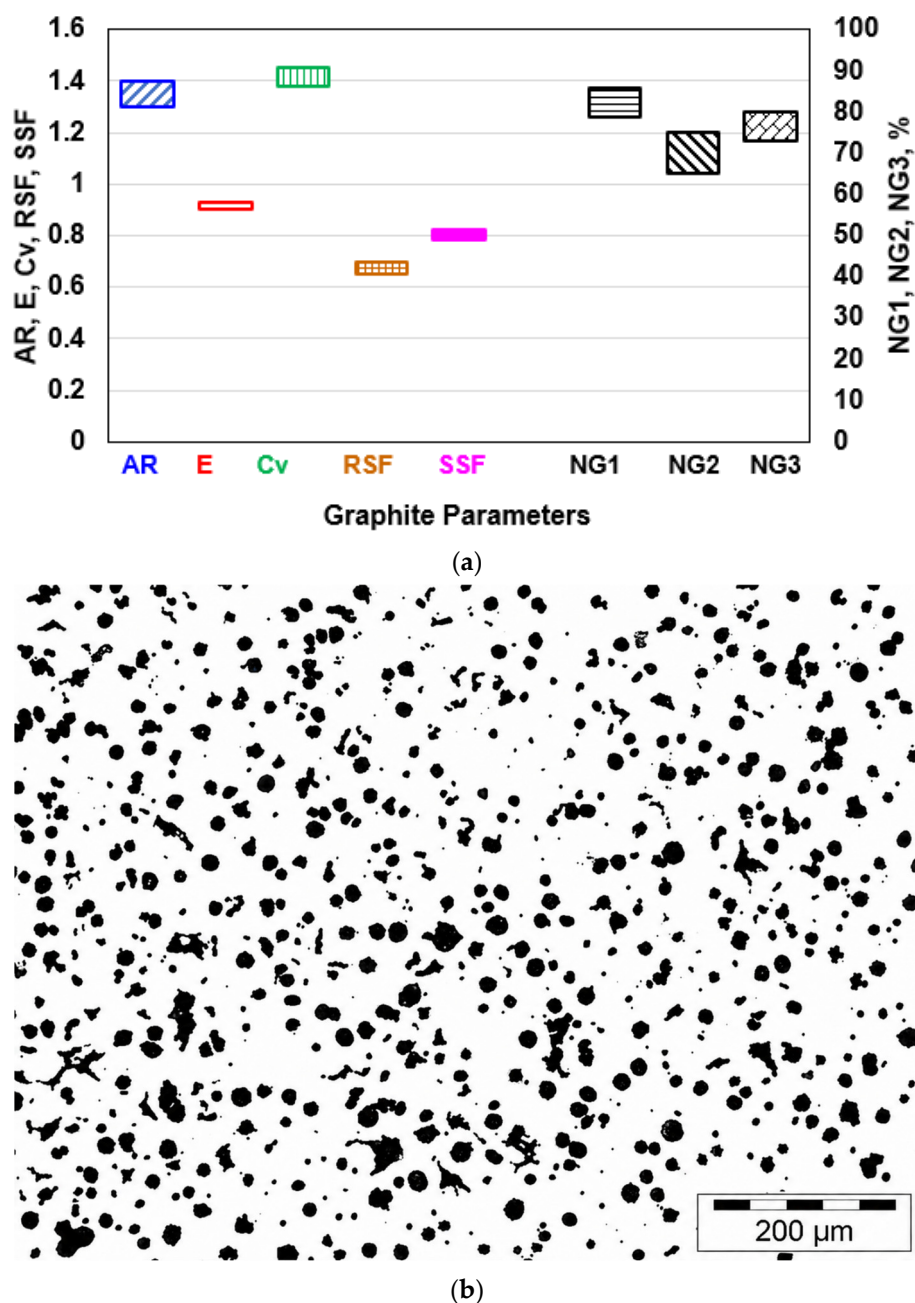


Figure 5. Graphite parameters for the entire range of wall thicknesses of wedge casting (3–15 mm) (a) and structure for the 15 mm wall thickness (b) of 4.55% Si ductile cast iron solidified in a sand mould (Program II).

Figure 5b shows a typical structure obtained at 15 mm wall thickness, characterized by the following average parameters: 464 nodules/ mm^2 , RSF = 0.68, SSF = 0.79, NG1 = 81.7%, NG2 = 65.3%, NG3 = 73% and NG = 69.1%.

Figure 6 shows the typical values for the graphite nodularity in wedge castings at different wall thicknesses, obtained by formulas stipulated in different international standards. The calculated graphite nodularity varied from NG1 (more than 80% levels) up to 65–70% (NG2), with intermediary values for NG3 and NG. Using the entire amount of spheroidal graphite Form V together with Form VI, the graphite nodularity according to the final (actual) standard (2019) increased with 4–5% compared to the initial variant (2015), which considered only 90% of the total area of Form V (ISO 945-1:2019) [31] graphite particles. The NG3 nodularity takes into consideration the reality of the presence of

the Slightly Irregular Spheroidal Graphite (Form V, ISO 945-1:2019 [31]; at higher real perimeter), at a high rate in high-Si ductile cast irons. This NG3 nodularity also appears to be more sensitive to solidification cooling rate variation.

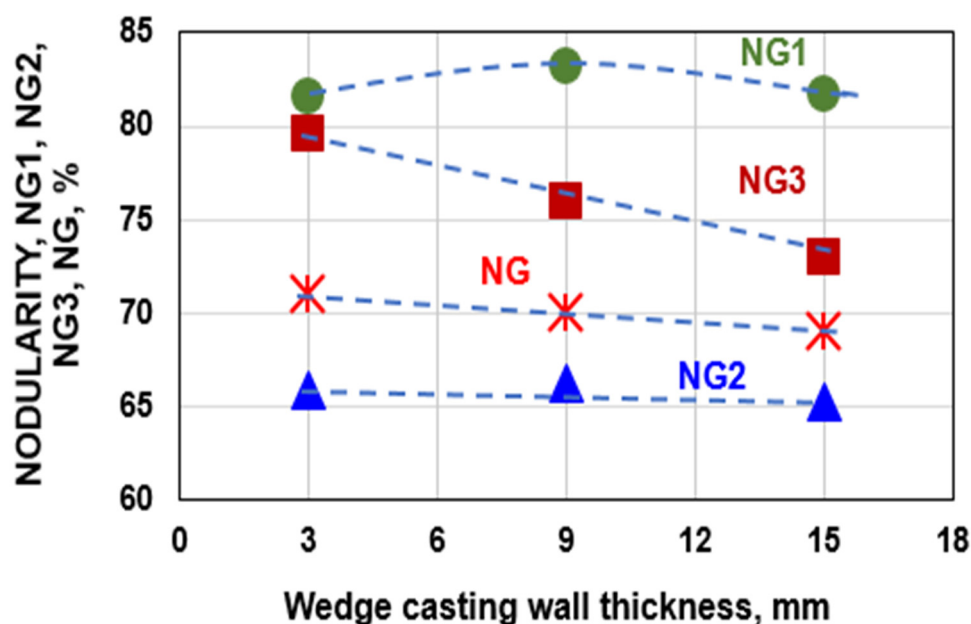


Figure 6. Graphite nodularity of 4.5% Si ductile cast iron solidified in a wedge casting in a green sand mould (Program II).

3.4. Metal Mould Versus Sand Mould Solidification

The structure characteristics of 4.5% Si un-inoculated ductile iron solidified in thin wall castings (up to 15 mm section size) obtained via a permanent (metal) mould (MM) versus a sand mould (SM) were evaluated [Program III] [24,25]. The two types of moulds are differentiated via their thermal diffusivity: $14,000 \text{ W}\cdot\text{s}^{1/2}/\text{m}^2\cdot\text{K}$ for the metal mould and $1396 \text{ W}\cdot\text{s}^{1/2}/\text{m}^2\cdot\text{K}$ for the green sand mould, resulting in a higher solidification cooling rate in the metal mould [16].

The graphite particles' real perimeter was 3–5% higher than the convex perimeter, while the values of these parameters were 41–43% higher in the sand mould. The increase in the casting section size favoured an increase in graphite perimeter, with it being much greater for the sand mould. The difference between the graphite particles' perimeter in the sand mould and metal mould for a given casting section size was higher for Pr than for Pc. For the shape factor involving graphite particles area and the maximum size, the metal mould solidification had a higher graphite compactness degree, as this factor had higher values than in the sand mould casting: $\text{RSF} = 0.68\text{--}0.7$ versus $0.59\text{--}0.64$. The wall thickness variation had a small effect on the sand mould, but practically no effect on the metal mould [24].

No free carbides and 70–100% ferrite in wedge casting solidified in the sand mould, and up to 15% carbides and 10–20% ferrite in the metal mould characterize the metal matrix. High-Si ductile cast iron solidification in a metal mould may be more sensitive to pearlite and carbide stabilizing elements than in a sand mould, which is documented to tolerate them. Nodule count is at a 30% higher level in a metal mould (75% nodules less than $15 \mu\text{m}$ size) and less depending on the wedge casting wall thickness. In a sand mould, only 37% of nodules are less than $15 \mu\text{m}$, while the increase of the section size led to a 30% decrease of the nodule count. The use of a metal mould led to higher average values for graphite nodularity compared to a sand mould. The effect of the wall thickness is more visible for

metal mould solidification: the higher the wall thickness, the higher the graphite nodularity obtained [25].

Typical structures obtained in sand mould (SM) and metal mould (MM) solidification are included in Figure 7, for 12 mm wedge casting wall thickness, at the following parameters (MM versus SM): AR = 1.295/1.32, RSF = 0.711/0.69, NC = 680/460 nodules/mm², NG2 = 80/75%, and NG3 = 79/74%.

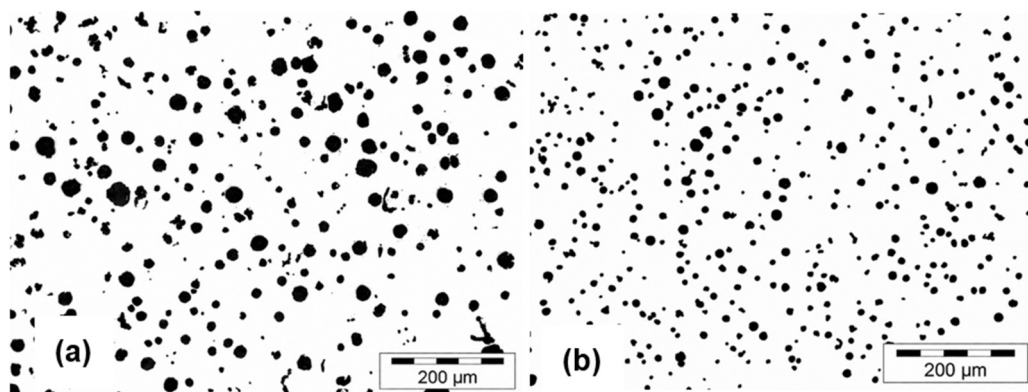


Figure 7. Typical structures at 12 mm wall thickness using a sand mould (a) or metal mould (b) (unetched condition) (Program III).

More data was obtained for the graphite particle size parameters, their representative shape factors, and graphite nodularity calculus, involving the real perimeter of the graphite particles (found to be sensitive to the variation of the solidification rate). Figure 8 summarizes the representative parameters of graphite, as average values of the solidification of wedge castings, measured by the wall thickness interval of 6–15 mm.

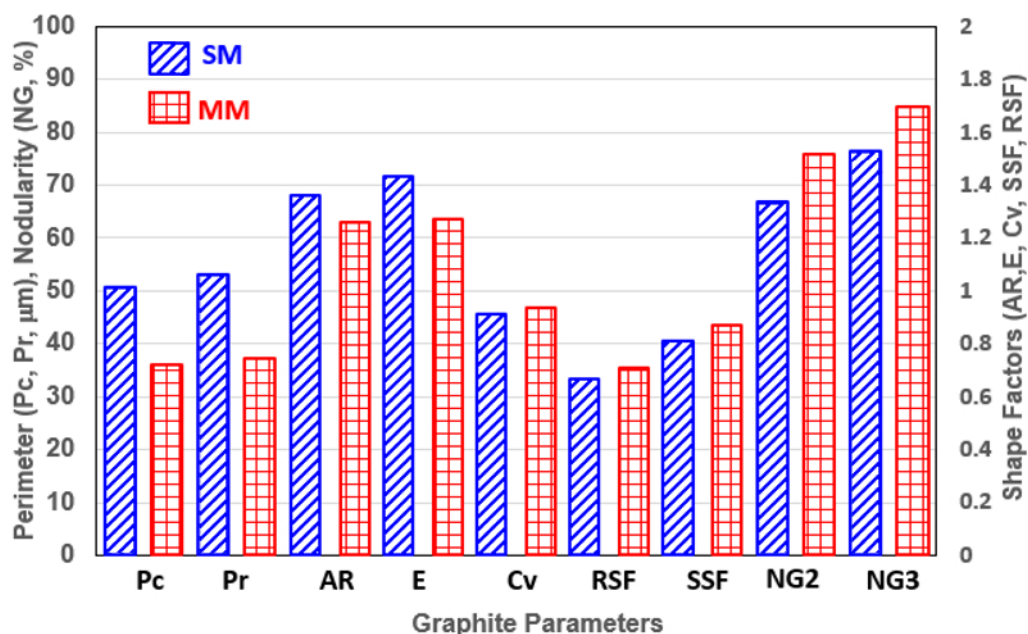


Figure 8. Average values of graphite parameters in wedge castings (6–15 mm thickness range) solidified in sand moulds (SMs) and metal moulds (MMs) (Program III).

The solidification at a high cooling rate, such as in the metal mould, improves the graphite parameters in high-Si ductile irons, comparing to the sand mould: lower values for both convex (Pc) and real (Pr) perimeter, aspect ratio (AR) and higher level of Roundness

(RSF) and Sphericity (SSF), which illustrates the higher degree of compactness of graphite particles closer to the spherical shape.

For both solidification conditions, the RSF parameter is part of the range that characterizes the graphite Form V (ISO 945-4:2019) [29], slightly irregular spheroidal graphite (see Figure 3), but at a higher level for MM. Consequently, the nodularity involving RSF has also improved (NG2, from 67 to 76% as average values).

Similarly, the shape factor involving real graphite particles perimeter (SSF) is also improved, with the increase of the corresponding nodularity (NG3) from 76.4% to 84.9%, as average values.

3.5. Solidification on the Metallic Chill

The presence of an external metallic chill in a resin sand mould influenced the graphite phase characteristics of high Si/SiMo alloyed ductile cast irons was evaluated (Program IV, 4.55% Si/5.25% Si/4.8% Si and 2.3% Mo; Irons 4, 5, and 6; Tables 1 and 2) [26].

The test samples, with 6.5×21 mm bases (the small one on the chill), 56 mm height and 60 mm length, were solidified in a furan resin sand mould on a cast iron external chill (12 mm thickness). The structure analysis [metallographic and image analysis] was recorded to evaluate ferrite, carbides and graphite amount, as well as the graphite size, count and shape forms parameters, on the sample height (3 parallel directions), up to 25 mm distance from the metallic chill (1 mm between the points analysed, 0.588767 mm^2 area each). Figure 9 shows the typical graphite structures of 4.5% Si and 4.8% Si and 2.3% Mo, un-inoculated ductile cast irons solidified in furan resin sand moulds [26].

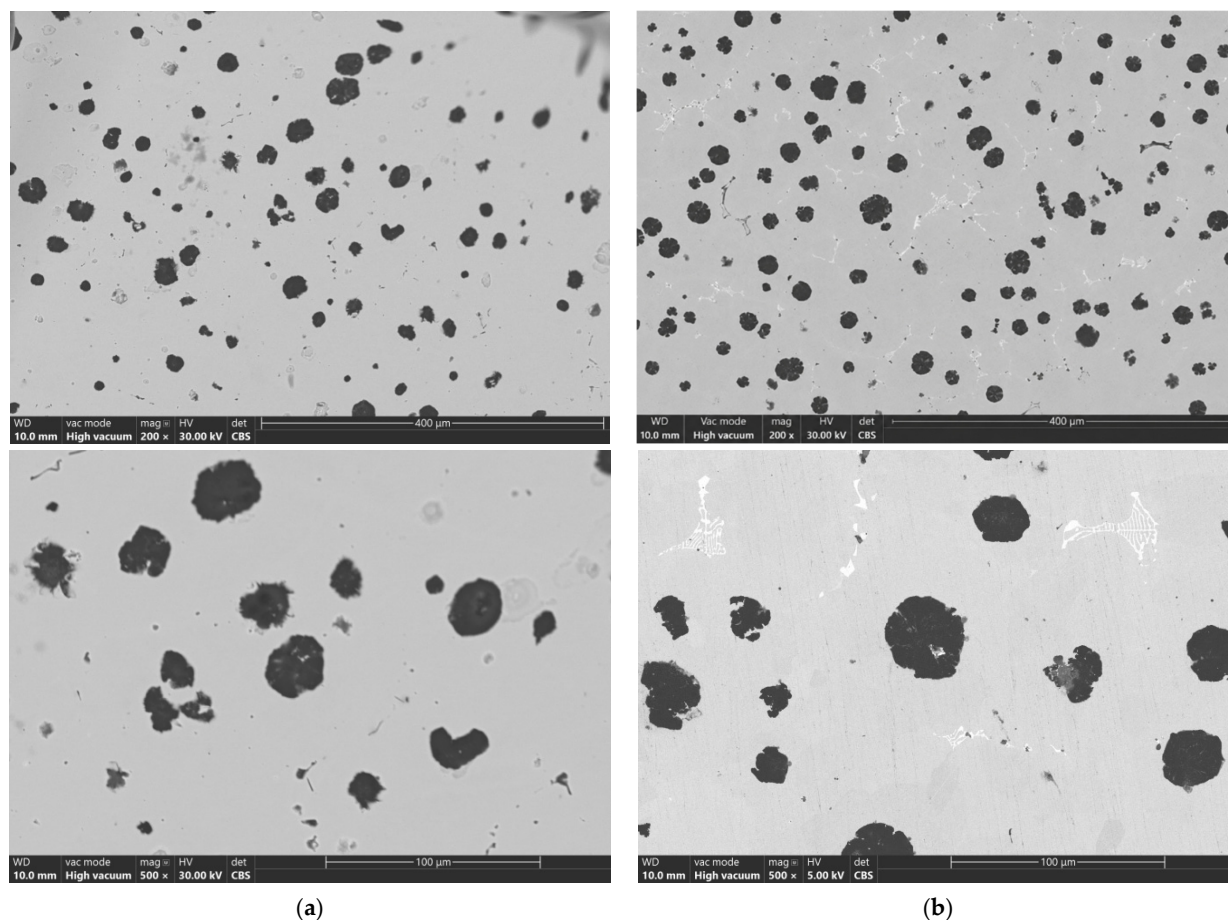


Figure 9. Graphite in 4.5% Si (a) and 4.8% Si and 2.3% Mo (b) ductile cast irons (unetched condition; SEM analysis; Program IV).

At 4.55% Si iron, a chilled zone of up to 6–8 mm resulted, with carbide presence, lower graphite amount, higher nodule count, and higher ferrite amount. Si increasing up to 5.25% Si led to a limited affected surface zone (up to 3 mm), characterized by no carbides and a limited graphite amount at the highest nodule count. Mo addition led to the most extensive chilled zone (11–15 mm) with the lowest amount of graphite and ferrite and the highest carbide occurrence, at the lowest nodule count [26].

Figure 10 summarizes the important parameters of graphite characterization obtained by the analysis of the ductile iron samples from 1 mm up to 25 mm distance from the metallic chill, as previously reported [26]. Any parameters, such as the minimum ($F_{\min}$) and maximum ($F_{\max}$) ferret and convex (P_c) and real (P_r) perimeter of the graphite particles are part of large ranges, which are more evident for Si–Mo alloying. For other parameters, such as the aspect ratio (AR), convexity (Cv), elongation (E) and complex shape factors, RSF and SSF, narrower domains resulted. The increase of the silicon content from 4.55% up to 5.25% visibly increased AR and E and decreased SSF and RSF, with the maximum detrimental effect for 4.8% Si and 2.3% Mo.

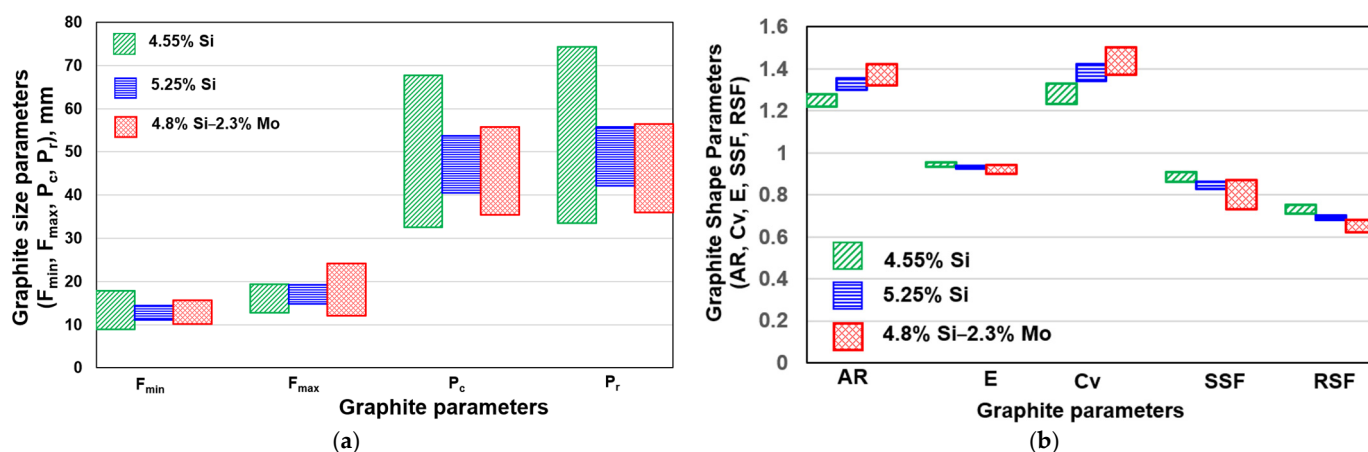


Figure 10. General range of the graphite parameters from 1 mm up to 25 mm distance from the metallic chill (Program IV): (a) size and (b) shape factors.

All test irons are characterised by Form V (ISO 945-4:2019) [29], slightly irregular spheroidal graphite morphology, typical for the roundness shape factor $RSF = 0.6$ – 0.8 range, at the highest position at 4.55% Si (0.71–0.75), intermediary at 5.25% Si (0.68–0.71) and the lowest position at 4.8% Si and 2.3% Mo (0.62–0.68). The chill solidification affects the graphite nodularity of 4.55% Si iron less, with the maximum influence for 4.8% Si and 2.3% Mo iron (nodularity decreasing with the distance from the chill) and by the imposed $RSF = \min. 0.80$, especially at 5.25% Si content [26].

More data was obtained on graphite characterization as effects of alloying grade and solidification cooling rate (it decreases with the distance from the metallic chill). The newly obtained data led to the graphs included in Figure 11, which show the variation of the graphite nodularity, NG1 (Figure 11a), NG2 (Figure 11b) and NG3 (Figure 11c), with the distance from the metallic chill, at different alloying grades of the test ductile cast irons.

There is a visible difference between the tested ductile irons, with nodularity decreasing from 4.55% Si to 5.25% Si iron and up to 4.8% Si and 2.3% Mo (the lowest level), for all the nodularity calculus formulas (Equations (3), (4) and (6)) and cooling rates.

According to the findings of the previously presented programs, NG1 (typically applied for compacted graphite irons) is the maximum nodularity, with a lower detrimental effect of Si-increasing and with appropriate positions of 5.25% Si and 4.8% Si and 2.3% Mo

irons at a higher solidification cooling rate (up to 16 mm distance from the metallic chill; after that the nodularity of Si–Mo iron decreases abruptly).

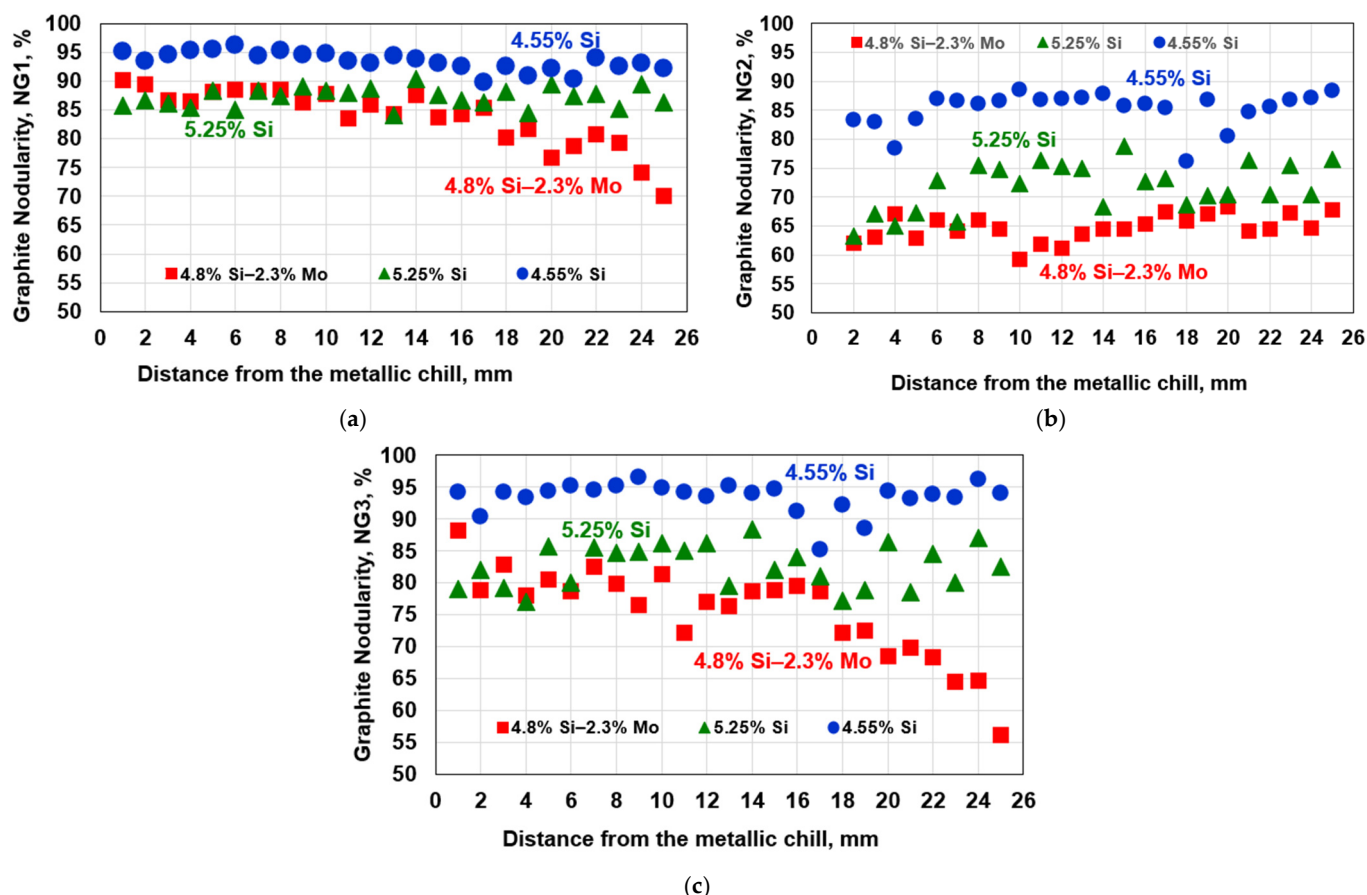


Figure 11. The influence of the distance from the metallic chill and the alloying grade on the graphite nodularity calculated with different formulas (Program IV): (a) NG1; (b) NG2; (c) NG3.

On the other hand, the evolution of the nodularity of 4.55% Si and 5.25% Si irons have a parallel aspect. NG2 nodularity, applied for ductile irons, has visibly lower values compared to NG1, with test ductile irons more differentiated, but in the same directions (the highest nodularity for 4.55% Si and lowest for SiMo iron). Nodularity is severely affected by the highest content of Si and SiMo alloying for the highest solidification cooling rate (up to 6 mm distance from the metallic chill).

Replacing the graphite shape factors (RSF with SSF) in the calculus of the graphite nodularity applied for ductile irons (NG2) led to NG3 nodularity. This nodularity is close to NG1 position for 4.55% Si and more appropriate for the other two irons.

The correlation between the aspect of structure and graphite parameters and different nodularity obtained led to the conclusion that the NG3 formula is better for characterizing graphite nodularity in ductile irons at these levels of silicon and solidification conditions.

3.6. The Influence of Inoculating Elements on the Graphite Particles Parameters

Inoculation is a metallurgical treatment meant to promote graphite formation instead of carbides, by sustaining the formation of graphite nucleation sites at a lower solidification undercooling degree. Inoculants are usually complex materials, including elements such as Si, Al, Ca, Ba, Sr, Zr, RE (rare earth), Bi, etc., able to interact with active elements in the molten iron (Si, S, N, C, O, etc.). The resulting compounds, with simple or complex chemistry (sulphides, oxides, oxy-sulphides, nitrides, carbides, etc.) will play the role

of the graphite nucleation sites, depending on the other influencing factors, such as the iron melting procedure and the chemical composition, holding time, pouring parameters, casting solidification conditions, etc. [32].

In the ductile iron production, the inoculation application is practically compulsory, in many cases, to avoid free carbides formation and the necessity to apply the heat treatment, respectively, and especially in critical conditions, such as a low content of silicon, thin wall castings, a mould with high heat transfer capacity, etc. On the other hand, inoculation also appears to be useful in improving the graphite phase quality in ductile iron, such as to increase nodule count, to improve nodule distribution, to increase nodule compactness degree, to promote nodule formation at the end of solidification (beneficial for decreasing micro-shrinkage formation), etc. [32].

Si-alloyed ductile cast irons, at 3.0–6.0% Si general range, are generally characterized by a high graphitization capacity, due to the contribution of a high silicon content, so they are not expected to allow the formation of free carbides or to require the application of inoculation for this reason, respectively. As an exception, very thin wall castings solidified in conventional conditions require a strong inoculation effect. For example, for 2.4–4.4% Si and 1–8 mm wall thickness castings, a ladle inoculation did not avoid carbides formation for less than 3 mm thickness, but a double inoculation (ladle + in-mould) led to free carbides castings at 2 mm wall thickness [33]. A higher solidification cooling rate, such as for a metal (permanent) mould [24,25,34] or using external metallic chill in the mould [26], also require inoculation to avoid free carbides. Specific inoculation necessity was identified to avoid chunky graphite formation [11–15,17–19] or porosity [19].

In ductile cast irons, the inoculation was also found to affect the quality of the nodular/spheroidal graphite particles, namely by increasing their compactness degree (approaching a sphere) [32].

The results of a research program (V, Tables 1 and 2) were recorded in order to compare the possible effects, from this standpoint, of the usual commercial inoculants used (Ca–FeSi, Ca,Ba–FeSi and Ca,RE–FeSi) by in-mould inoculation of a ductile iron at 4.5% Si content [16,25]. Wedge casting W3 (ASTM A 367 [35]), solidified in a resin sand mould, was used to evaluate structure characteristics at different wall thicknesses (3–15 mm).

Nodule count appears to be less influenced by inoculation and inoculating elements than by section size (cooling rate) variation, with a more restricted values range compared to other parameters. A Roundness Shape Factor at the 0.6–0.8 level illustrates the common formation of slightly irregular spheroidal graphite morphology (Form V, ISO 945-4:2019) [29], more accentuated for thin wall thicknesses (less than 0.7 for less than 10 mm section size), with Ca, RE–FeSi inoculation at the lowest values, for the entire section size range. By involving the real perimeter, higher than the convex perimeter, Sphericity Shape Factor (0.33–0.78) is lower than Compactness Shape Factor (0.75–0.85), with the same large range distribution for more than 3 mm section size, like the one obtained for the real perimeter. Ca,Ba–FeSi is better than Ca–FeSi inoculation, while Ca,RE–FeSi drastically decreases this graphite shape factor [16].

Different relationships between the values of the representative graphite particles size parameters ($F_{\max}$, A_G , P_c and P_r) were found, resulting from the wedge casting wall thickness variation (from 3 up to 15 mm) and different inoculation variants applied (Ca/Ca,Ba/CaRE). A good relationship between the area (A_G) and the maximum size ($F_{\max}$) of the graphite particles was found, without a visible influence of the inoculating elements. Convex perimeter (P_c) increases with the increase of the graphite particles area and their maximum size, generally in a good relationship, with P_c – $F_{\max}$ as performance, and without visible differentiation in the applied inoculating elements. The graphite particles' real perimeters are higher for higher area and their maximum size, but with different positions

of the inoculating elements system. There is a good relationship between $Pr-A_G$ and $Pr-F_{max}$ for Ca–FeSi and Ca,Ba–FeSi inoculated ductile cast irons [25].

New data was obtained as the relationship between complex graphite shape factors (RSF, SSF) and size characteristics of graphite particles (A_G , Pr). Figure 12 shows the relationship between the Sphericity Shape Factor (SSF) (Equation (2)) and the two important graphite particles parameters, area (A_G) and real perimeter (Pr), for the three systems of applied inoculants. Graphite particles had 100–270 μm^2 area and 40–70 μm real perimeter in these inoculated ductile cast irons. The considered shape factor (SSF) had different values depending on the inoculating system: 0.77–0.85 for Ca,Ba, 0.75–0.81 for Ca and 0.33–0.78 for Ca,RE. It appeared that Ca,Ba–FeSi was better than simple Ca–FeSi, as regards the graphite shape factor level, while Ca,RE–FeSi led to the lowest shape factor values, dispersed in a wide area.

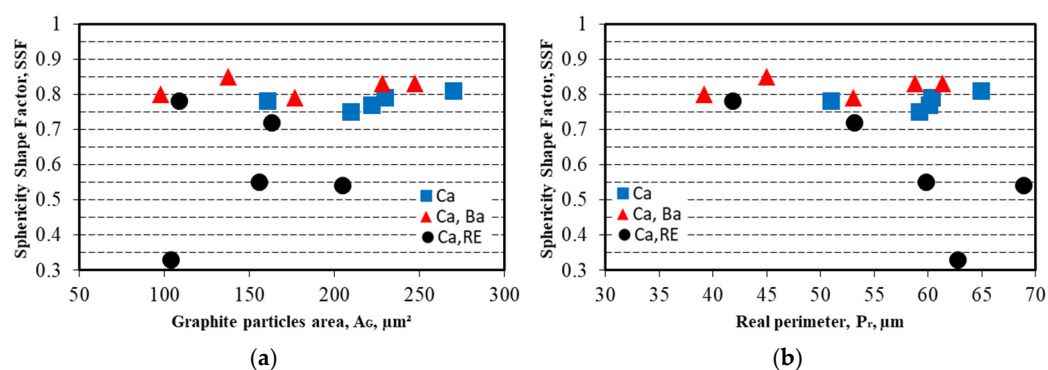


Figure 12. Relationship between Sphericity Shape Factor (SSF) and graphite particles area (a) and real perimeter (b) depending on the inoculating elements (4.5% Si ductile iron) (Program V).

It could be concluded that in the existing experimental conditions, for high Si-ductile iron solidified in thin wall castings, Ca,Ba–FeSi inoculation appears to be better than simple Ca–FeSi for most of the graphite parameters to improve, while Ca,RE–FeSi has the lowest beneficial effect, especially as it negatively affects the compactness degree of graphite particles.

4. Conclusions

- (1) The occurrence of chunky graphite and the negative effect on the compactness degree (approaching a sphere) of spheroidal graphite particles characterize Si-alloyed ductile cast irons (DI) (especially at more than 4% Si).
- (2) Slightly irregular spheroidal graphite morphology (Form V, ISO 945-4:2019) [29] characterized by roundness shape factor $RSF = 0.59–0.75$ was found to be typical for Si and SiMo ductile irons, in all of the test conditions, such as Si level (4.2–5.25% Si), Si–Mo system (4.1–4.8% Si + 1.6–2.3% Mo), mould type (green sand, resin sand, metal mould, external metallic chill), un-inoculation and inoculation, and inoculating element type.
- (3) The increase of the Si-content negatively affects the compactness degree of spheroidal graphite particles (transition from VI to V form).
- (4) The increase of the solidification cooling rate, typical for the low wall thickness, metal mould or metallic chill solidification improved spheroidal graphite morphology, expressed by higher graphite shape factors values.
- (5) Inoculating elements had influence on the graphite shape factors in high-Si DI, solidified in thin wall castings, where a Ca–Ba system was better than simple Ca and Ca-RE could promote graphite particles at high real perimeter and lower shape factors.

- (6) By the application of stipulations of the initial nodularity evaluation standard ISO/WD 945-4-2015 [28] (including RSF and considering 100% Form VI and 90% Form V graphite), lower values resulted for graphite nodularity, as NG2 = 65–87% in all the present tests.
- (7) The final (actual) nodularity evaluation standard for ductile iron (NG, ISO 945-4-2019) [29], applying Roundness Shape Factor (RSF) and considering 100% graphite particles forms V and VI (ISO 945-1-2019) [31] increased nodularity (NG) 4–5% compared to the initial standard in this field (NG2).
- (8) ISO 16112:2017 [27], applied for Compacted Graphite Iron (CGI), recommends a graphite nodularity formula, including the same RSF, but which systematically led to higher values for nodularity, NG1 = 77–97%. The difference between NG1 and NG2 (NG) depends on the Si content and cooling rate.
- (9) By replacing the shape factor RSF (involving graphite particles area and their maximum Ferret) with SSF (involving area and real perimeter) in the nodularity formula stipulated by ISO/WD 945-4-2015 [28], the nodularity NG3 = 72–95% resulted, which is intermediary between NG1 and NG2 (NG).
- (10) In all the expressed formulas, the graphite nodularity is higher for higher solidification cooling rate, and lower for higher Si level, reaching the lowest level for Si–Mo irons (specifically at high Mo alloying grade, 1.6–2.3% Mo).
- (11) The higher the imposed graphite shape factor in the nodularity calculation, the lower the resulting graphite nodularity and higher the negative effect of Si-alloying (the highest is on the occurrence of regular spheroidal graphite (Form VI)).
- (12) The correlation of the aspect of structure, graphite parameters and nodularity led to the conclusion that the nodularity formula according to ISO/WD 945-4-2015 [28] (including 100% total area of Form VI and only 90% area of Form V), with SSF instead of RSF as graphite shape factors, is recommended for characterizing nodularity in High-Si/SiMo Ductile Cast Irons (especially for more than 4% Si).
- (13) Both recommendations, referring to the participation of Form V (ISO 945-1-2019) [31] graphite (90% instead of 100%) and graphite shape factor (SSF instead of RSF) must be validated by mechanical properties, based on numerous research results obtained in this respect. This will be a future research activity.
- (14) The obtained data could sustain specific stipulations to be introduced in ductile iron standards, referring to the graphite structure and nodularity for high Si/SiMo materials, and the necessity to approach specific metallurgic treatments able to increase the Form VI graphite rate to obtain these ductile irons, including Mg-treatment and/or inoculation procedures.

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