Microstructural Features and Mechanical Properties of Compacted Graphite Iron Treated With Calcium-Magnesium Based Masteralloy

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(Submitted 27 September 2002; in revised form 26 February 2003)

This paper presents some data on the microstructural features and mechanical properties of as-cast compacted graphite iron (CGI), produced through treatment with a special Ca-CaC_2-Mg masteralloy. Information on graphite morphology and matrix features, obtained using different numerical indices evaluated through a computer-based image-analyzing system and SEM observation of deep-etched specimens, shows that the graphite form is a mixture of ASTM types I, II, and III (compacted graphite), interspersed in a matrix with a 2:1 ratio of pearlite to ferrite. An average hardness (BHN) of 219, Charpy-V-Impact energy of 7.92 J, tensile strength of 354.5 MPa, and a corresponding elongation of 1.20% has been obtained for the as-cast iron. The fracture surfaces of tensile and room temperature impact tested specimens showed mixed modes of fracture.

Keywords compacted graphite iron, graphite morphology, graphite nodulariser/modifier, microstuctural features and mechanical properties

1. Introduction

With respect to structure and/or physico-mechanical properties, compacted (or vermicular) graphite iron (CGI) is a grade between grey and ductile (or nodular graphite) irons.[1,2] This versatile and latest member of the cast iron family has been developed to provide an iron that did not need extensive alloying but would be of higher strength than grey iron, while having better thermal conductivity and machinability than ductile iron. This combination of desirable properties has resulted in the use of CGI for applications in lightweight, high-strength automobile structures and components exposed to thermal cycles or subject to possible thermal shock.[3-5] It has been suggested that the relatively short span with rounded edges and rough surfaces of the graphite particles improve adhesion with the iron matrix, increasing, relative to the grey iron, the tensile strength by at least 75% and stiffness by 35% while roughly doubling fatigue strength.[6] Furthermore, the inherent graphite continuity provides good thermal conductivity and vibration damping.

The increased development of CGI for use as cylinder blocks, exhaust manifolds, brake and head power-train components, coupled with planned CGI high-volume-modular-manufacturing-systems (HVMMS) programs from car and truck manufacturers,[7-9] has allowed Ford to achieve aggressive targets for engine performance, size, weight, and cost that could not simultaneously be met by traditional engine materials, such as aluminum or alloyed grey cast iron. The engine satisfies Euro (2005) emissions requirements and, assisted by the stronger CGI engine material, has the potential to be equally compliant with Euro V (2008) legislation while satisfying mass production requirements and economics.[9]

A problem in the production of compacted graphite and ductile irons is in the use of expensive and dangerously reactive magnesium or the also expensive but less effective cerium, as graphite nodularisers/modifiers. Conventionally, CGI is produced via the ductile iron nodularizing route, but with a more stringent control or a very narrow process window with magnesium or cerium under-treatment to achieve a required degree of compaction in a given section of a component. While the use of magnesium alone in graphite spheroidizing under-treatment (to produce CGI) presents practical difficulties in controlling "fading" and overcoming the environmental effects accompanying its addition to molten iron, cerium has a powerful chilling effect, dross formation, and graphite flotation problems. CGI is also known to form when ductile iron fades or deteriorates with the latter resulting from the introduction of small amounts of anti-spheroidizing elements such as titanium with magnesium and/or cerium. However, the incorporation of such anti-spheroidizing elements suffers from the disadvantages of segregation, section sensitivity, a tendency to form spheroids in thin sections, and contamination of returns.[10] Another method used for graphite compaction involves nitrogen,[11] usually introduced through the use of nitrided ferromanganese (80% Mn, 4% N). Disadvantages of N treatment include difficulty in producing uniform structures in castings with varying section size and the occurrence of unsoundness and fissure defects with excessive N.

Thus, the ever-growing interest of metallurgists in research efforts aimed at finding cheaper, safer, and equally effective substitutes to the above mentioned treatment agents is understandable. Recent research interest in the use of multi-element treatment agents in composite with magnesium and/or cerium[12-14] is predicated on the need to overcome the practical difficulties in controlling the treatment process, nodulariser-

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modifier sensitivity, fading, casting defects, and accompanying environmental effects in their individual use. The work reported in this paper was part of a comprehensive program of research to gauge the effectiveness and establish the optimal treatment requirements of a series of calcium-magnesium based graphite nodularisers/modifiers for the production of compacted graphite and ductile irons. This report summarizes the microstructural features and properties of the optimum CGI produced using a special Ca-CaC_2-Mg masteralloy.

2. Experimental Procedure

The alloying content and optimum concentration of the nodularizing/modifying pre-alloy composite were chosen based on data from earlier work.[14,15] In the pertinent case, laboratory grade Ca, CaC_2, and Mg content in the masteralloy was fixed at 10%, mixed with Fe45Si (Fe-45.00Si-0.30Al-0.25Ce) and compacted into capsule-shaped briquettes. The optimum CGI was produced with a pre-calculated Ca/CaC_2/Mg ratio of 1:1:3 at an approximate effective Ca:Mg ratio of 1:1.846 and at 3.00-wt.% treatment addition to the melt. A heat of a base iron of composition (Fe-3.73C-0.98Si-0.10Mn-0.023S-0.054P-0.04Cr-0.20Cu-0.005Mg) was melted in an induction furnace, superheated to ≈ 1550 °C, held for 3 min, and tapped at 1480 °C onto a calculated amount of the treatment agent in a preheated treatment ladle and cast after graphitization inoculation with 0.5% Fe75Si (Fe-73.29Si-2.64Al), without reladling. The castings were produced in green sand moulds. Testpieces for spectrographic analysis were also poured.

Standard testpieces for microstructural and mechanical properties testing were machined from the parallel and rectangular portion of the Y blocks. Tensile testpieces were 8 mm in diameter with a minimum gauge length of 40 mm and with threaded shoulders, prepared in accordance with DIN EN 10.002 Zugversuch. Notched Charpy impact testpieces were 10 × 10 mm in cross section and 55 mm in length, with a 2 mm deep V notch prepared in accordance with DIN 50 115/04.91 Kerbschlagbiegeversuch. Appropriate specimens were also prepared for micro-hardness testing. Tensile tests for all specimens were carried out at a constant displacement rate of 0.05 mm/s on a Heckert Tira 2300 (Starragheckert GmbH, Chemnitz, Germany) tensile testing machine equipped with autographic recording of the stress-strain curve, to allow determination of the 0.2% offset yield strength, as well as the tensile strength and elongation at failure. Notched Charpy impact tests at room temperature were carried out on a Heckert WPD Psd300 automatic impact instrument with a striking energy of 300 J. The impact energy (J), lateral expansion (mm), and percentage fibrous area (%) were obtained. Following standard procedure, a series of Vickers micro-hardness measurements on specific phases in specimens was conducted using appropriate accessories on a Neophot 2 (Carl Zeiss, Vienna, Austria) microscope.

Composition was analyzed using both EDX and WDX spectroscopy run via a Kevex 4850s microscope interface module on a Kevex Delta (Carl Zeiss, Vienna, Austria) class analyzer. Carbon and sulphur were analyzed using a coulometric method. Information on graphite morphology and matrix features was obtained using a computer-based image analyzing system and Macros III (Carl Zeiss, Vienna, Austria) software. Both global and feature specific parameters for graphite were programmed for evaluation using universally accepted definitions. A detailed assessment of structure (matrix features and graphite morphology) was made on samples from the broken impact test specimens, since these were representative of the structure of the sections used for the determination of the mechanical properties. Each of these measurements was made on 25 different fields and averaged. Fracture surfaces were examined using Zeiss Jenaphot 2000 (Zeiss Jena, Vienna, Austria) and DSM960 (Zeiss Jena, Vienna, Austria) digital scanning electron microscopes coupled to an image analyzing system.

Table 1 Chemical Composition (wt.%) of CGI Produced

C	Si	Mn	S	P	Cr	Cu	Mg	Ca	Al
3.65	2.35	0.12	0.008	0.026	0.030	0.25	0.50	0.10	0.15

3. Results

3.1 Microstructural Characteristics

The chemical composition of the CGI Y block casting produced is given in Table 1. Table 2 presents data on the microstructural features of the iron in the as-cast state. Approximate ratio of graphite, pearlite, and ferrite is 2:7:4, respectively, with a negligible level of other phases (usually, cementite, ledeburite, phosphide eutectic, etc.). Table 3 presents data on graphite morphology using various numerical assessment indices.[17,18] The graphite structure corresponds to a mixture of ASTM types I, II, and III (compacted graphite) evaluated according to ASTM A247. Figure 1(a) and (b) illustrates SEM observations on the form of the graphite where the matrix has been etched away. Figure 1(b) at a larger magnification illustrates a typical example of the thick, round-ended graphite particle co-existing alongside graphite spheroids. Generally, the iron has a sizeable proportion of nodules, quasi-nodules, and a predominance of vermicular graphite particles interspersing the structure.

3.2 Mechanical Properties

The results of mechanical properties measured for the iron in the as-cast state are given in Table 4. Nominal values for the hardness were 180 (ferrite) and 242 (pearlite), giving an average hardness of 211. The mechanical properties measured for this iron are typical of commercial CGI. Figure 2(a) and (b) show representative fractographs of tensile and room temperature impacted specimens, respectively, of the iron. Figure 2(a) shows a typical fracture surface where a thick short-spanned, round-ended (compacted) graphite particle is shown unbroken, between two mix-mode fracture surfaces. The matrix in both tests showed mixed degrees of deformation (brittle as well as ductile fracture), quantified by the percent fibrous (ductile) fraction, FF (Table 4).

4. Discussion

The results of this investigation show that the special multi-material Ca-CaC_2-Mg composite in treatment aggregates the additive effect of known graphite nodularisers and/or modifi-

Table 2 Microstructure Data for the As-Cast CGI

Graphite Area, %	Pearlite Area, %	Ferrite Area, %	Other Phases Area, %
15.64	55.05	28.80	Trace

Table 3 Graphite Parameter for the As-Cast CGI, Determined Using Image Analysing System

Parameter, Index	Mean Value
Area, %	15.64
Particle count, N	135
Particle size, ($\times 10^{-3}$ mm^2)	1.58
Nodularity, optimum; %	60.15
Excursion ratio E	0.63
Form factor, aspect ratio	0.57
Degree of spheroidization	0.55

Table 4 Mechanical Properties of the As-Cast CGI

	Tensile Properties (a)			Charpy-V-Impact Values (b)		
Micro-Hardness, Hv 0.05	$Rp_{0.2}$, MPa	R_m, MPa	E_m, %	Φ, J	LB, mm	FF, %
219	257	354.5	1.20	7.92	1.22	15.35

(a) $Rp_{0.2}$, 0.2% offset yield strength; R_m, tensile strength (mean); E_m, elongation (mean)
(b) ϕ, Charpy V-Impact energy; LB, lateral expansion; FF, fibrous (ductile) fraction

ers, particularly calcium and magnesium, which can be used at a much lower treatment agent requirement and relatively more clement treatment conditions. Although this study has not examined in detail the graphite modification role(s) or the mechanism(s) of modification of this composite, it is clear from comparing data between the compositions of the base/treatment materials and the iron produced (Table 1) that a significant reduction, notably in sulphur and phosphorous contents (as high as 65% and 50%, respectively), was achieved. This supports the fact that the removal or outright neutralization of sulphur and phosphorous in the iron melt through reactions with the modifier(s) is indeed necessary for the production of compacted graphite and ductile irons. Sulphur, phosphorous, and to some extent oxygen are known to be surface active. By reducing the graphite/iron interfacial energy, their segregation on the graphite/iron interface and selective adsorption on the graphite prism planes can promote the extended interfacial characteristics of flake graphite.[19,20] The modifiers ensure that the graphite basal planes would now have the lower surface energy in contact with the molten iron, resulting in the formation of nodular and/or compacted (due either to insufficient or under-treatment agent level or degeneracy resulting from over treatment) graphite. On the basis of OM, TEM, and SEM studies on CGI, Guilemany and Llorca[20] have suggested an identical rapid growing plane for both spheroidal (ductile) and compacted graphite particles (see Fig. 1a and b, where both compacted and spheroidal graphite particles are seen to coexist). According to Guilemany and Llorca,[20] in the presence

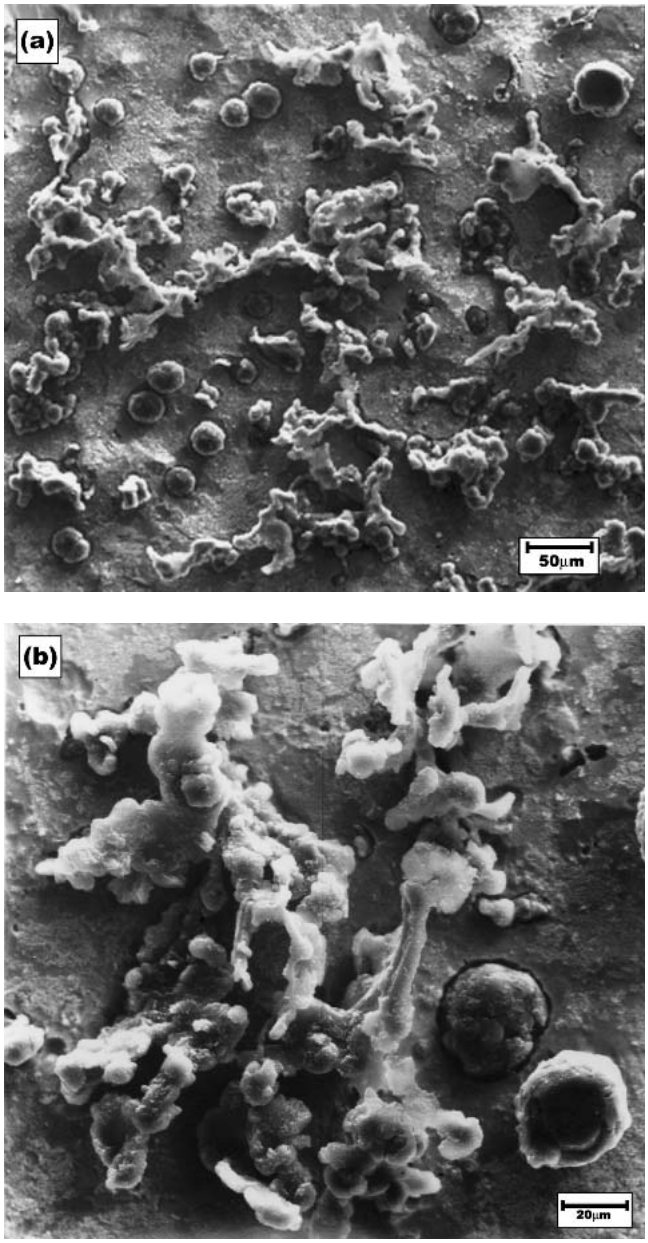

Fig. 1 Scanning electron micrographs of as-cast specimens with matrix etched away to show graphite form: **(a)** ×200 (50 μm), **(b)** ×500 (20 μm)

of modifiers, the graphite in CGI develops a facility for growth in the maximum "compacity" plane in a direction perpendicular to the basal plane of the graphite hexagonal crystal. In contrast to the spheroidal, this growth of the compacted plane is not uniform, and so the development of the characteristic worm-like form is favored in spite of being in the same layer and through special directions.

In the present Ca-CaC_2-Mg composite formulation, magnesium is the better-known treatment agent, for which the desulphurization-nodularization/modification mechanism is well reported. Calcium and calcium carbide on the other hand are used most often as deoxidants, dephosphorisers, and desulphurisers in the iron and steel industry. Calcium and calcium containing

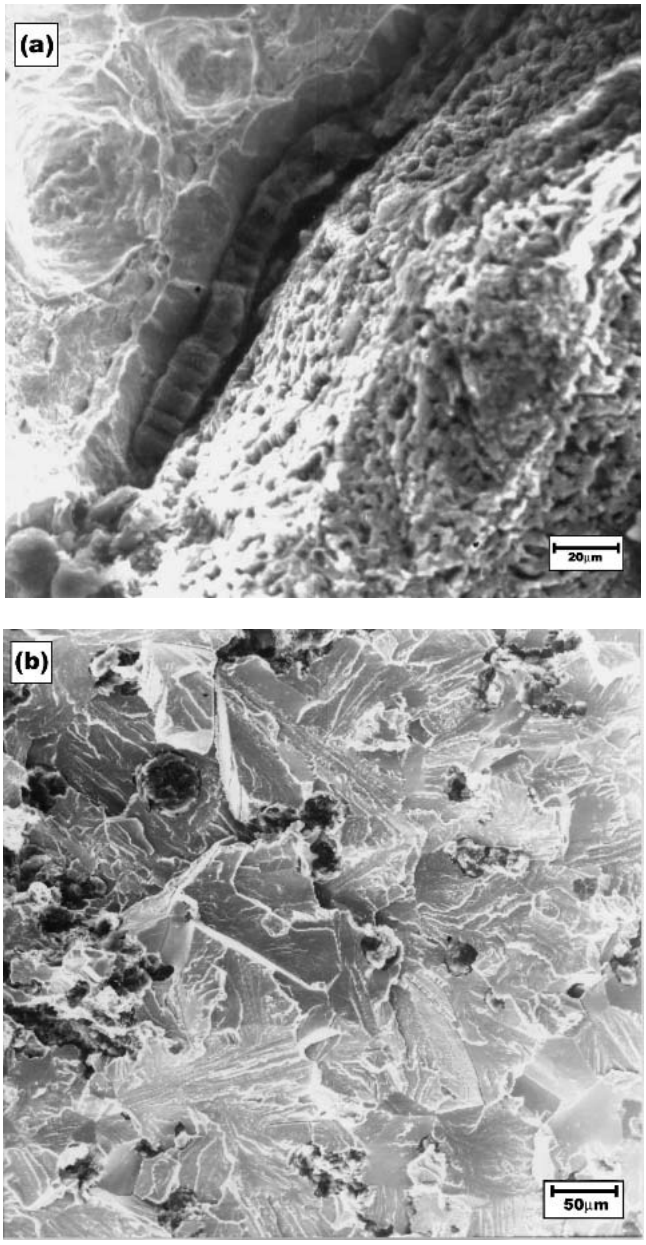

Fig. 2 Typical fractographs of specimens: **(a)** tensile tested; ×500 (20 μm), **(b)** impact tested; ×200 (50 μm)

compounds have been successfully used as modifiers before in-mould nodularizing treatment with magnesium.[21,22] Calcium is now attractive due to its dual and simultaneous role in reducing sulphur content to the desired level and hence its efficacy as a nodulariser and/or modifier based on the above-mentioned mechanism. In most of these cases, calcium has been found to be more efficient in the form of compounds and/or when introduced to the molten bath through specially designed modes.[16,23,24] The explanation of Imasogie et al.[14,16] on the effectiveness of calcium in this regard should suffice here.

It is known that the percentage of spheroidal versus compacted graphite, indicated here by the form factor (aspect ratio) and to some extent, the degree of spheroidization (Table 3) in CGI, affects its tensile strength in particular. The greater the number of graphite particles approaching a spherical shape, the higher the relative tensile strength.[2] Thus, both tensile strength and elongation increase as the percentage of spheroidal graphite increases. This is also to be expected for a matrix that is about 2:1 ratio of pearlite to ferrite, with little or no brittle or hard phases, as in the pertinent iron's case. The level of graphite spheroidization in CGI is critical to its consideration for application in functional components and in applications where some level of toughness is required. The iron in the present case compares favorably with the recently reported Intermet Corporation's[8] (Troy, MI) new product, the Enhanced Compacted Graphite Iron (ECG) meant for use in the DaimlerChrysler new 4.7 L, V-8 engine introduced in the 1999 Jeep Grand Cherokee. This "winning" and "production feasible" iron has increased nodularity of up to 50% and casts soundly without risers, to obtain a high mold yield. Thus, it is to be expected that the present iron with a form factor or aspect ratio of 0.57, a degree of spheroidization of 0.55 (Table 3), and the reported levels of mechanical properties (Table 4) would adequately fit such a bill.

5. Conclusion

A special Ca-CaC_2-Mg masteralloy has been used in a graphite modifying treatment of a base gray cast iron melt to produce a mixture of ASTM types I, II, and III compacted graphite iron (CGI) with a matrix of ratio 2:1 of pearlite to ferrite. The treatment advantages include lower modifying agent cost and relatively hard or brittle phase free matrix microstructure, resulting in an iron with comparable properties to that produced via a standard or commercial treatment route.

Acknowledgments

The author gratefully acknowledges the technical assistance of Professor H. Blumenauer and the staff of the Institut für Werkstofftechnik und Werkstoffprüfung, Technische Universität Magdeburg, Germany. The author also thanks Professor O.O. Adewoye, Chief Executive Officer, Engineering Materials Development Institute, Akure, Nigeria, for his interest and valuable discussions.

References

1. Anon: "Cast Iron Grades," Guldsmedshytte Bruks AB, http://www.guldsmedshyttebruk.se/eng/castiron_grades.htm (8 Aug 2002).
2. CSD: "Tech. Articles. Mechanical Properties of Compacted Graphite Iron," http://www.castingsource.com/tech_art_graphite.asp (8 Aug 2002).
3. Anon: "Compacted Graphite Iron, Technical Data," I. H. Dick & Co. Ltd., http://www.thdick.co.uk/ductile.htm (8 Aug 2002).
4. H. Wong: "Techniques Being Developed to Produce High Strength, Lighter Weight Automobile Castings," *Metal Casting,* http://www.oit.doe.gov/IOF/metalcast/ (8 Aug 2002).
5. G.S. Vasilash: "Material Driving Powertrain Production: Compacted Graphite Iron," http://www.autofieldguide.com/columns/gary/0102mat.html (8 Aug 2002).
6. G. Georgiou: "Machining Solutions for Compacted Graphite Iron," Machine Shop Guide Web Archive – Jan/Feb. 2002, http://www.machineshopguide.com/pdf/Jan02/compact.pdf (8 Aug 2002).
7. Ford Motor Company: "Ford to Introduce Compacted Graphite Iron (CGI) Cylinder Block," http://www.dieselnet.com/news/0107ford.html, 2001 (8 Aug 2002).

8. Anon: "Ductile Iron News, Intermet Leads Joint Development of Enhanced Compacted Graphite Iron," http:/www.ductile.org/magazine/1999_1/compacted.htm, 1999 (8 Aug 2002).
9. Anon: "Tupy Noticias On Line, Ford Selects Tupy and SinterCast for New Engine Programme," http://www.tupy.com.br/ing/pagina_news_29_06_2001i.htm, 2001 (8 Aug 2002).
10. R. Elliott: "An Introduction to Cast Irons" in *Cast Iron Technology,* Butterworths, London, 1988, pp. 20-24.
11. F.A. Mountford: "The Influence of Nitrogen on the Soundness and Structure of Grey Cast Iron," *Brit. Foundryman,* 1966, *59,* p. 141.
12. M.A. Rice, A.B. Malizo, and H.F. Brooks: "The Use of Multiple Nodularizing Elements in Making Ductile Iron Pipe," *Trans. Am. Foundrymen's Soc.,* 1974, *82,* pp. 15-26.
13. Anon: "Compacted Graphite Iron, Tech. Info. 13, Revision No. 2," Elkem ASA, Silicon Division, Oslo, Norway, Mar 1997.
14. B.I. Imasogie, J.A. Ali, and A.A. Afonja: "Properties of Ductile Cast Iron Nodularised with Multiple Calcium-Magnesium Based Master Alloy," *Mater. Sci. Technol.,* 2000, *16*(2), pp. 194-201.
15. B.I. Imasogie: "Development and Characterization of Isothermally Heat-Treated Nodular Cast Irons," Ph.D Thesis, Obafemi Awolowo University, Ile-Ife, Nigeria, 1994.
16. B.I. Imasogie, J.A. Ali, and A.A. Afonja: "Properties of As-Cast and Heat Treated Nodular Graphite Cast Irons, Melts Treated with CaSi-CaF_2 Alloy," *Scand. J. Metall.,* 2001, *30*(2), pp. 91-102.
17. Y. Tanaka, H. Saito, I. Tokura, and K. Ikawa: "Relationship Between Some Physico-Mechanical Properties and Numerical Indexes of Graphite Shape in Compacted-Vermicular Graphite Cast Irons," *Trans. ASME,* 1982, *104,* pp. 60-65.
18. A.G. Fuller, P.J. Emerson, and G.F. Sergeant: "Report on the Effect upon Mechanical Properties of Variations in Graphite Form in Irons Having Various Amounts of Ferrite and Pearlite in the Matrix Structure, and the Use of NDT in Assessments of Mechanical Properties of Such Irons," *AFS Trans.,* 1980, *88,* pp. 21-50.
19. S.E. Franklin and R.A. Stark: "Application of Secondary Ion Mass Spectroscopy to Study of Graphite Morphology in Cast Iron," *Met. Sci.,* 1984, *18,* pp. 187-200.
20. J.M. Guilemany and N. Llorca: "Metallographic Differences Between Compacted Graphite Cast Iron and Vermicular Graphite Cast Iron," *Pract. Met.,* 1984, *21,* pp. 299-306.
21. A.M. Skrebtsov and L. A. Bol'shakov: "Control of the Properties of High-Strength Cast Iron by Inoculants of Different Composition," *Liteinoe Proizvod,* 1981, *1,* pp. 30-31.
22. A.M. Petrichenko, V.A. Priymak, and M.V. Mozharov: "Use of Complex Inoculants for Production of Spheroidal Graphite Cast Iron," *Liteinoe Proizvod,* 1981, *1,* pp. 7-8.
23. H. Kajoka: "Metal Desulphurization by Calcium-Carbide Injection" in *3rd McMaster Symp. on Iron and Steel Making, External Desulphurization of Hot Metals,* McMaster University, Hamilton, ON, Canada, May 1975, pp. 22-23.
24. K. Kaneko, N. Sano, S. Takenchi, and Y. Matsushita: "Desulphurization and Dephosphurization of Cr-Fe and Si-Cr-Fe Alloys by CaC_2-CaF_2, CaO-CaF_2 and CaSi-CaF_2 Fluxes Using Plasma Jet Remelting" in *Proc. 3rd Int. Iron and Steel Cong.,* ISS-AIME, Warrendale, PA, 1978, pp. 583-88.