# GRAPHITE PHASE FORMATION IN AI-ALLOYED DUCTILE IRONS

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Abstract The influence of aluminum, within the range of 0.08 - 6.16 wt.%, on the microstructure and hardness of as-cast ductile cast iron has been studied. A number of irons of different composition have been made by green sand casting and gravity die casting of appropriate design to provide the experimental materials. It has been found that Al addition and higher cooling rates in permanent mould casting both promote pearlite formation in the as-cast irons. Detailed microscopic observation revealed that foreign particles (with a high concentration of Al, Si and Mg) within the graphite spherulites probably behaved as nucleation sites, and that Al addition can promote spheroidal graphite nucleation and growth. Furthermore, it was illustrated that spherulites with a fan-like structure consist of aggregates of graphite platelets and interplatelet regions.

Key Words Ductile Cast Iron, Spherulite, Graphene, Aggregation Layers, Foreign Particles, Pearlite Formation

چکیده در این تحقیق اثر آلومینیم در محدوده ۲/۱۰-۶/۱۹ درصد وزنی بر ریز ساختار و سختی چدنهای داکتیل ریختگی مطالعه گردیده است. نمونه های چدنی با ترکیب شیمیایی متفاوت توسط ریخته گری در قالبهای ماسه ای تر و ریژه با طراحی مناسب برای انجام آزمایشهای بعدی ساخته می شوند. ملاحظه می گردد که افزودن AI و زیاد کردن سرعت سرد کردن از طریق ریخته گری در قالبهای دائمی هر دو باعث افزایش تشکیل پرلیت در چدنهای ریختگی می شوند. مشاهدات میکروسکوپی با جزئیات بیشتر نشاندهنده وجود ذرات غریبه (دارای غلظت بالای AI) هم (هم و و و گرافیت های کروی می باشد که احتمالاً همانند محلهای جوانه زایی عمل کرده است. بنابراین افزایش AI می تواند جوانه زایی و رشد گرافیت کروی را بهبود دهد. علاوه بر این نشان داده شده است که گرافیت ها با ساختار پره ای شکل متشکل از لایه های متناوب از صفحات گرافیت و نواحی بین صفحه ای می باشند.

## 1. INTRODUCTION

The nodular irons are relatively inexpensive and special properties such as a high degree of ductility and shock resistance are achieved by modifying the form of the graphite. The success of ductile iron means that it currently accounts for about 20 to 30% of the cast irons used in most industrialized countries [1].

The microstructure of nodular cast irons is normally a ferrite-pearlite matrix containing a random distribution of graphite. The two most important factors affecting the graphitization, and the number and size of graphite nodules, are composition and cooling rate [2,3].

The final microstructure of the as-cast ductile iron is related to the melting process and alloying elements and determined by the heat treatment. Satisfactory iron production depends on a technical understanding of the as-cast condition of irons.

The ability to change the morphology of graphite in cast iron from flake to nodular form was originally developed by Morrogh in 1947 [4].

This structure can be produced in the as - cast state by treatment with additions such as magnesium, or cerium and magnesium, to the molten iron. These ductile irons are known as nodular or spherical (spheroidal) graphite irons (SG) [2,3,5]. Ductile cast iron has higher strength and toughness than grey cast iron with flake graphite form. Deviation from nodular graphite to flake graphite introduces lower strength because the flakes act as easy crack paths and stress concentrators in the metal. By modifying the graphite to spheroidal form a higher level of tensile strength and ductility can be achieved [2,3].

The presences of well-distributed graphite in the matrix, and a relatively high volume fraction of pearlite, are responsible for the good mechanical properties of the ductile irons including excellent wear resistance. It is now well known that the noticeable properties of irons directly depend on the volume fraction of graphite in the matrix.

Graphitizing elements modify the formation of graphite and amongst these elements silicon is the most important. Composition of irons is an effective factor to increase or decrease the nucleation and/or growth of graphite. Some elements, for example, Si, Al, Ni, Cu, and P enhance the graphitization potential, and Mo, Cr, and V have the opposite effect [3]. Zhukov [6] reported that silicon and aluminum are the strongest graphitizing elements in cast irons and initially show the same behavior. However, these elements do present some differences. He explained that the graphitization theory is very complicated and the thermodynamics may determine the similarity or differences in the behavior of Fe-C-Si and Fe-C-Al systems.

DeFrancq et al. [7] published the first study relating the complete substitution of aluminum for silicon in cast iron. According to Walson [8], the Al-alloyed cast iron has advantages in applications for light weight components with high strength at room and elevated temperatures, higher resistance to thermal shock, better graphitizing tendency and higher resistance to oxidation at high temperature. It has been described that Al acts as an inoculant and refiner in gray cast iron and increases the number of eutectic cells. It was reported that Al addition favors tendencies for graphite nucleation in cast irons [9].

Boutorabi [1991] reported that fully ferritic and or pearlitic matrices could be obtained in both as cast or heat-treated condition in SG Al iron. It has been found that the graphite nodule number is much greater than in corresponding SG iron [10]. According to previous study, the tribiological behavior of 2.2% Al-alloyed austempered ductile iron in comparison with Si-alloyed irons has been studied [11]. They argued that the influence of Al on bainitic reaction is similar to silicon in conventional SG cast irons. The result has shown that Al is a strong graphitizer and produced high wear resistance.

It is well documented that graphite may forms directly from the melt during solidification. It is generally accepted that the starting point for solidification takes place on a nucleus with different chemical composition.

The crystal structure of graphite is hexagonal. Semi-infinite hexagonal layers, which are sometimes named "graphene", are stacked with - ABAB- order. Double and Hellawell [12] explained that during solidification, the initial hexagonal monolayers or grapheme sheets, which are carbon precipitated from molten metal can undergo further development. They believed that in a clean iron, the extension of graphene leads to tangles, or wrapping convolutions, resulting in spherulitic graphite. The wide range of possibilities in folding, wrapping and branching of the layers can determine the different graphite forms reported.

Atoms can add much more easily in the -a-directions as compared with the -c- direction. Double and Hellawell [13] explained that if the layer lattice is rolled to a sphere, the possibility of molecular addition in the -c- direction will be increased. They believed that the hexagonal monolayers or graphene sheets freely precipitate from the molten metal solution and reported that impurities in the melt such as oxygen and/or sulfur would promote the growth of graphite sheets in the -c- direction, which leads to the flake graphite form.

Baihe Miao et al. [14] illustrated many fan-like structures, which are aggregates of graphite platelets forming a spheroid of graphite. In addition, Baihe Miao et al. [15] suggested, based upon the Double and Hellawell model, a graphite

TABLE 1. Composition of Irons, wt-%.

Alloy	С	A1	Si	Ni	Mn	P	S	Mg	Fe
0.08%A1	3.71	0.08	1.08	0.03	0.09	< 0.005	< 0.005	0.05	Balance
0.48%A1	3.68	0.48	1.06	0.04	0.06	< 0.005	< 0.005	0.05	44
0.55%A1	3.67	0.55	1.13	0.05	0.06	< 0.005	< 0.005	0.05	44
1.71%AI	3.58	1.71	1.18	0.04	0.07	< 0.005	< 0.005	0.05	4.6
1.82%AI	3.56	1.82	1.20	0.34	0.09	< 0.005	< 0.005	0.05	44
2.11%Al	3.55	2.11	1.21	0.04	0.11	< 0.005	< 0.005	0.06	44
3.10%A1	3.48	3.10	1.24	0.05	0.10	< 0.005	< 0.005	0.06	44
4.88%AI	3.44	4.88	1.22	0.05	0.10	< 0.005	< 0.005	0.05	44
6.16%Al	3.25	6.16	1.35	0.07	0.10	< 0.005	< 0.005	0.06	44

spherulite consisting of conical helixes, in which the {0001} planes of graphite within each conical helix grow out of the graphite platelets. They believed that crystallographic defects present in the graphite structure could change normal growth of the spherulite and lead to bending or branching of the crystal within the basal plane. The interplatelet area illustrates that the spiral growth of graphite is not perfect.

### 2. EXPERIMENTAL

Experimental ductile irons with the composition range given in Table 1 were produced in a Morgan gas - fired furnace (with 25 kg capacity lift - out crucible) and a high - frequency-melting plant of 20 kg capacity (with a tilting crucible). After melting, the iron was superheated at 1550°C and

small pieces of solid aluminum added by plunging into the bottom of the liquid metal. Enough time was given to dissolve the aluminum completely in the molten metal. Following aluminum treatment, FeSiMg (5%Mg) alloy was plunged into the liquid iron and ejection of molten metal during solution of magnesium prevented by use of special enclosed reaction vessels. Finally, post-inoculation of ferro-silicon containing 75%Si was carried out in the crucible. The sandwich technique was used to treat the melt of each iron with a ferro-silicon alloy containing 5%Mg at 1400°C. In the casting of ductile irons, late addition of inoculants into the melt is much more effective and can be achieved by an in-mould technique. As shown in Figure 1 a reaction chamber which holds the granulated inoculant in the running system was required to ensure a uniform flow of inoculant over the whole

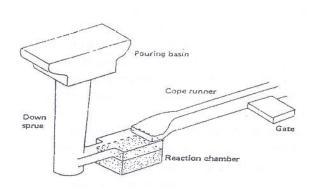


Figure 1. An in-mold spheroidizing gating arrangement for spheroidal irons [3].

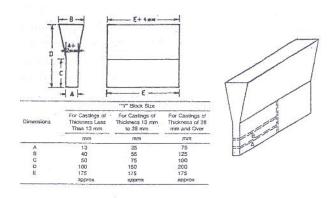


Figure 2. The standard dimensions of Y-block, in mm and two blanks for unnoched Charpy specimens (ASTM A 897M-90).

of the casting.

The melts were cast with a pouring temperature of approximately 1350°C by both green sand molding and gravity die-casting. Standard 12.5 mm and 25 mm Y-block sand moulds and permanent moulds were used as illustrated in Figure2; bottom gating introduced the metal smoothly into the mould cavity to ensure a sound casting. Quantitative measurements of the carbon content in the experimental irons were made using equipment at Swinden Technology Center of Corus Group PLC (formerly British Steel Ltd.). Analyses were performed by an infrared absorption method after combustion in an induction furnace and oxidation to CO2. Atomic absorption spectrophotometer (AAS) of Hi-Search Technology (HIST) at Birmingham University was used to analyze the aluminum content of high-Al ductile irons.

The compositions selected resulted in more than 90% nodularity of graphite. Optical microscopy, and scanning and transmission electron microscopy were used to show the microstructure of the spheroidal graphite (spherulite). Additionally, the test samples were examined by EDX to reveal the alloying element concentrations inside and outside of the graphite nodules.

For scanning electron microscopy, a Cambridge Series 3 SEM fitted with a Link 860 series 1 EDX system and a Cambridge Series 4 SEM were used. For the characterization of microstructure a working distance between 20-24 mm was chosen, and an accelerating voltage of 20kv and spot size between 4 - 6 nm.

The successful preparation of TEM thin foils from cast iron is more complicated than most kinds of metal alloys and steels, because there are two quite different phases (iron and graphite) in cast iron. When Jet polishing is used, a dark surface due to strong oxidation and corrosion can be obtained and the detachment of large graphite nodules can take place before adequate thinning.

A more laborious process of mechanical polishing and ion thinning as follows only achieved the preparation of a thin foil from the bulk samples:

 specimens were cut to sections approximately 250μm in thickness using a Struers Accutom-2 slitting machine

- 2) discs of 3mm diameter were punched from these sections
- 3) the 3mm diameter disks were mechanically polished to 80-100  $\mu$  m thickness
- the central specimen area was dimpled to 10-20 μm thickness using a Gatan precision dimple grinder model 656 and
- 5) the samples were ion beam thinned, initially under a 15° inclination angle and finally 10, for times ranging from 2 hr to 24 hr depending on initial thickness, using a Gatan dual ion milling machine model 600 beam thinner and a Gatan precision ion polishing system model 691 PIPS<sup>TM</sup> V4.31

The thin foils were examined using a Philips CM20 TEM operating at an accelerating voltage of 200 ky.

Identification of the structure and the phases present in the experimental specimens was determined with series of LM and SEM images. In determining the volume fraction for graphite, point counting was carried out on polished and unetched samples. Volume fraction of ferrite and pearlite in the matrix was measured by the same technique on polished and etched samples.

#### 3. RESULTS

Figure 3 show a typical microstructure of the iron containing 1.71%Al, in which the volume fraction of graphite present in the iron is about 12%. Figure 4 shows SEM micrographs of the experimental ductile irons with 0.48% and 4.88%Al content. As can be seen the graphite nodules are dispersed randomly in the microstructure (Figures 3-5).

The experimental irons in this investigation also showed good nodularity for different levels of aluminum content (Figures 5 and 6), whereas Al is classified as deleterious to spheroidization [3].

The results indicated that the number of graphite nodules increases with Al addition because of the strong graphitization effect of aluminum, while the graphite nodule size decreases for higher Al concentration (Table 2 and Figure 5).

The influence of composition and cooling rates on the structure and properties of as-cast ductile iron with aluminum contents in the range 0.08 to

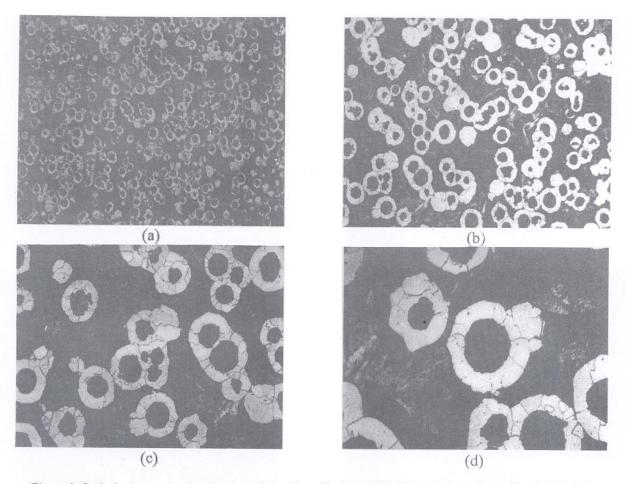


Figure 3. Optical micrographs showing the nodules of graphite in a 1.71% Al ductile iron after etching in 2% nital. This section was taken from a sample cast in a sand mould; (a) 50X, (b) 100X, (c) 200X, (d) 400X.

6.16 wt-% are shown in Table 2 and 3. The results show an increase in the number of graphite nodules, pearlite fraction and hardness, with an increase in aluminum content. This was accompanied by a reduction in the size of graphite nodules and lower ferrite content. In addition, the higher cooling rate induced by casting in a permanent mould compared with a sand mould, probably due to the increased under-cooling, also gave a similar result, i.e. an increased number of graphite nodules (Table 2) of smaller size, and increased hardness. However, there are some fluctuations in these parameters.

SEM-EDX analysis was carried out to describe the formation of the graphite nodules in the experimental irons. A cross-section of a graphite spherulite is shown in Figure 5. The

examination showed foreign particles in the graphite spherulites that contained high amounts of magnesium, aluminum, silicon and iron (Figure 5).

EDX examination showed that the foreign particles in the spherulite are compounds containing magnesium, aluminum, silicon and iron higher than in the surrounding graphite. A SEM micrograph near the edge of a graphite nodule in the 4.88%Al addition ductile iron is shown in Figure 8 (a). Figures 8 (b-e) are elemental EDX line scans of Mg, Al, Si and Fe across the area shown.

Figure 9 shows TEM micrographs from a 4.88% Al ductile iron at two different magnifications. Figure 9 (a) illustrates three graphite nodules at low magnification. Some

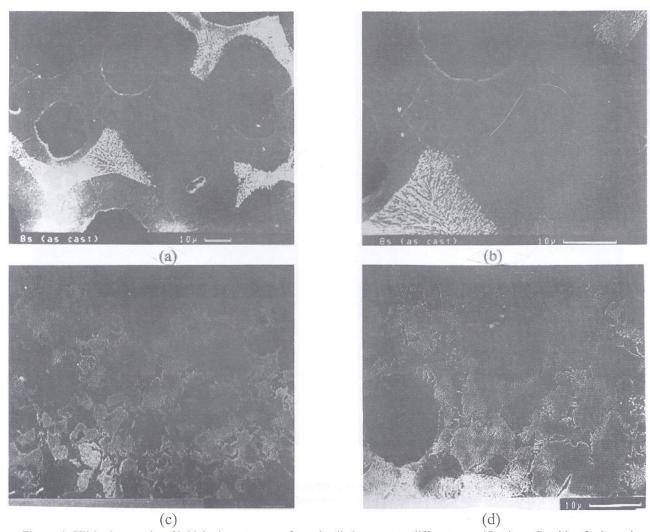


Figure 4. SEM micrographs of initial microstructure of two ductile irons at two different magnifications. Graphite, ferrite and pearlite are present in the structure (etched 2% nital); a and b, 0.48% Al and c and d, 4.88% Al.

nodule cross-sections were thin enough for TEM examination of the graphite structure. A bright field image of a section at high magnification is shown in Figure 9 (b). The structure of the spherulite is built up by aggregation of graphite platelets to produce a fan-like structure. This structure consists of graphite platelets and interplatelet regions. This observation is consistent with previous studies performed on two kinds of nodular silicon irons [15,16]. Figure 8 shows another TEM micrograph of the cross section of a graphite nodule. The bright field image illustrates the layered morphology of the graphite. The corresponding dark field image indicates that the

layers are in similar orientation.

# 4. DISCUSSION

The structure and properties of as- cast irons are dependent on numerous factors, one of the most important being composition which must be controlled during melting. Also, the form and distribution of graphite are associated with the composition [2,3]. In producing the experimental ductile cast irons careful control was necessary to achieve graphitization and to keep the variation of

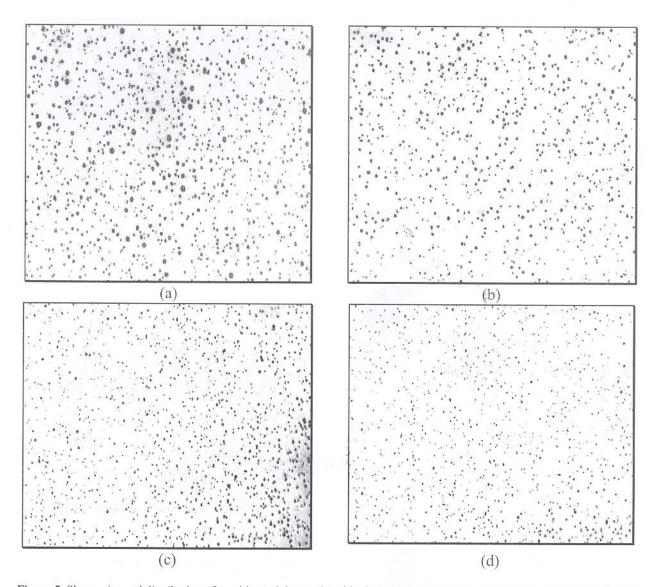


Figure 5. Shape, size and distribution of graphite nodules produced in the as-cast state by addition of aluminum; unetched, light micrograph; (a) 0.48% Al (X50); (b) 1.71% Al (X50); (c) 4.88% Al (X50); (d) 6.16% Al(X50).

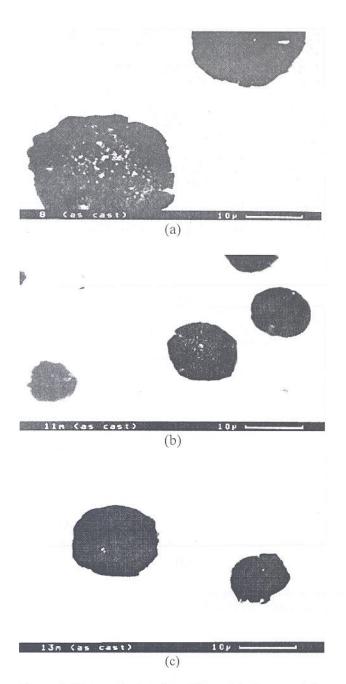
elements within the required range. It is well known that deviation from a spheroidal graphite shape leads to a serious reduction in the properties of irons, such as tensile strength and impact resistance.

The results obtained in the present work show that there is a significant increase in the graphite nodule number with an increase in Al content. The above finding is similar to those in Al ductile irons [10]. It is reported that large nodule number in SG Al iron can be attributed to the combination of a highly effective inoculants used and to the relative

difference in the rate of diffusion of carbon in liquid iron associated with aluminum.

Graphitization is a complex interaction between all variables and chemical composition on a macroscopic and microscopic level. Alloying elements are essential to provide sufficient treatment and aluminum content effect the transformation response for a given alloy.

It is suggested that Al relatively increases the carbon diffusion rate in the liquid iron because the nodule growth occurs at a higher freezing temperature. The same interpretation could be



**Figure 6**. Shape and size of graphite nodules in as-cast Alalloyed ductile iron, unetched (SEM); (a) 0.48% Al, (b) 4.88% Al and (c) 6.16% Al.

applied to explain the reduction of eutectic carbides in reduction of eutectic carbides in SG Al iron, the growth of which is favored at lower freezing temperature [10].

Hardness values appear to correspond to specific changes in the microstructure, which have

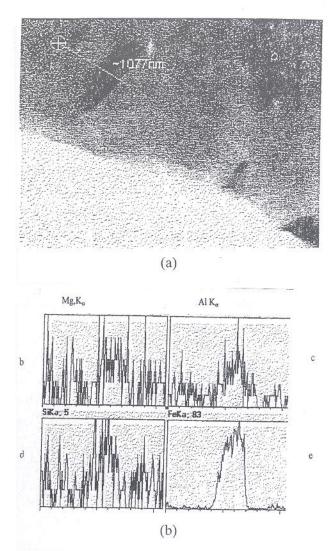


Figure 7. Electron micrograph in a 4.88% Al content ductile iron and the corresponding elemental EDX line scan in this area; (a) the microstructure of a section of graphite spherulite with several inclusions in the spherulite, (b, c, d and e) the magnesium, aluminum, silicon and iron elemental EDX line scans.

been identified in the present work. In another study, the effects of austempering variables on the hardness strength and ductility of Fe-C-Al iron with a spheroidal form of graphite, describes as SG Al iron is investigated. It has been shown that changes in the mechanical properties can be related to the morphology, size and volume of transformation constituents [17].

It is well documented that sulfur is a harmful element and that flake graphite is the dominant

TABLE 2. Microstructural Characteristics of As-Cast Experimental Irons Solidified in Different Mould Casting.

Alloy	Nodule count / mm <sup>-2</sup>		The ave	erage nodule size (µm)	Hardness (HV 50)	
	S	Р	S	Р	S	Р
0.08% Al	336	431	31.3	25.8	198.0	298.1
0.48% Al	339	438	29.4	25.0	197.8	288.5
1.71% Al	491	587	17.6	15.8	183.0	315.0
2.11% Al	484	594	16.3	14.9	184.6	322.9
3.10% Al	431	581	24.3	15.7	192.4	350.6
4.88% Al	472	563	17.2	14.4	246.9	341.2
6.16% Al	443	554	16.1	13.2	319.4	334.0

S: Sand mould casting

P: Permanent mould casting

TABLE 3. Structure and Phases for Al - Alloyed Iron Cast in Different Moulds.

Alloy	Structure	on sand mould	casting	Structure on permanent mould casting			
	Graphite, %	Ferrite, %	Pearlite, %	Graphite, %	Ferrite, %	Pearlite, %	
0.08% Al	13.8	76.4	9.8	13.1	47.1	39.8	
0.48% Al	13.9	75.8	10.3	13.0	45.3	41.7	
1.71% Al	12.1	24.7	63.2	10.7	21.6	67.7	
2.11% Al	11.8	23.7	64.5	9.9	18.8	71.3	
3.10% Al	9.8	21.2	69.0	7.8	13.7	78.5	
4.88% Al	9.9	10.5	79.6	7.6	8.4	84.0	

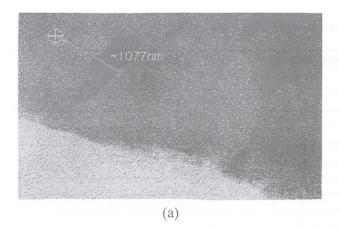
morphology in the presence of this element. It is also reported that S.G iron with less than 0.01%S can be obtained in the presence of Al, which acts as a desulfurizer [3,15].

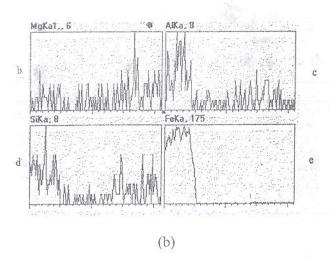
The difference in the number, size and distribution of graphite nodules occurs for different Al addition involving the nucleation and growth of new phase/phases in the prior liquid phase. It is believed that clustering of solute atoms on the prior phase to form zones or nuclei for heterogeneous nucleation depends upon the Al concentration and consequently the smaller size and larger number of graphite nodules (grains) for higher Al contents is a consequence of lower under-cooling which is necessary for heterogeneous nucleation and/or the possibility of larger amounts of nuclei created during the nucleation processes [18].

Particles can be observed within the spherulite which must be entrapped during growth of the

graphite from liquid iron. The growth of the spheroid is limited after envelopment by austenite [3]. It can be seen that around the spherulite the matrix is rich in Al. It is known that reactive elements including Mg, Ce and rare earths that promote spheroidal growth can adsorb on the graphite/liquid interface [3].

This indicates that graphitization could be improved by the aluminum concentration. In agreement with this study, it is found that aluminum in the range 0-0.1% acts as an inoculant and increases the number of graphite nodules [9]. In another study performed by Baihe Miao et al. [15] it was shown that cerium-rich nodules are present in the middle of most graphite nodules in irons modified by cerium additions. They believed that inclusions of Ce<sub>2</sub>O<sub>2</sub>S and Ce<sub>2</sub>O<sub>3</sub> formed when cerium or cerium and magnesium was added to alloys. It has been suggested that heterogeneous nucleation of graphite is associated with that part





**Figure 8**. Electron micrograph from a 4.88% Al content ductile iron and the corresponding elemental EDX line scan in this area; a) the microstructure of a section of graphite spherulite, (b, c, d, e) the magnesium, aluminum, silicon and iron elemental EDX line scans.

of the inclusions that are not removed in the slag and remain in the melt. They have illustrated that cerium-rich inclusion which acts, as heterogeneous nucleation sites are present in most graphite nodules in irons treated by cerium.

In addition, segregation causes a non-uniform transformation response within the material on a microscopic level. Weis suggested that no one specific substance is responsible for nucleation, but rather that all suspensions present in the melt might have a nucleation effect [19].

It is also indicated that the tiny bubbles in the





**Figure 9.** TEM bright field images of graphite spherulites in 4.88%Al ductile iron at different magnification (a) three graphite nodules are illustrated at low magnification and (b) typical structure observed inside the graphite nodule of Figure 9a.

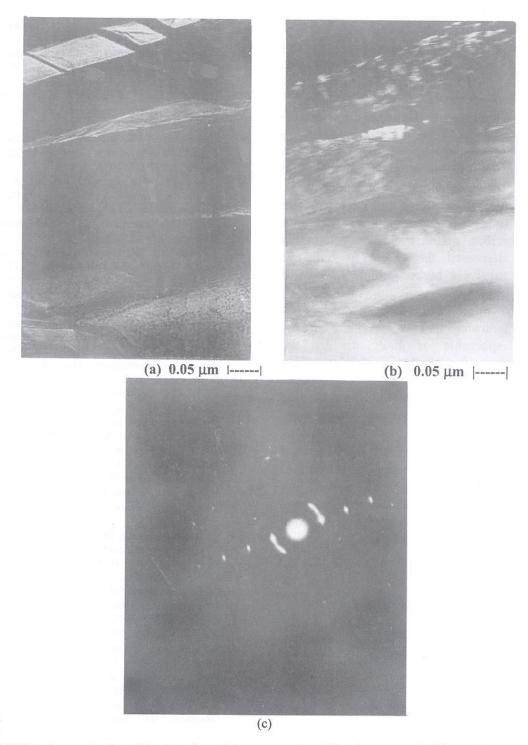


Figure 10. TEM micrographs of graphite spherulite and the corresponding diffraction pattern; a) BF image, b) DF image and c) corresponding diffraction pattern.

liquid metal, created by using a variety of alloying elements are ideal sites for nuclei that supply the energy levels for the growth of graphite.

# 5. CONCLUSIONS

Examination of the as-cast Al-alloyed ductile irons

resulted in the following conclusions:

- At the different concentrations of Al employed, more than 90% nodularity was achieved with the nodules dispersed randomly in the microstructure.
- There is a reduction in the average nodule size with increasing Al content, but equally, a higher nodule count with increasing Al, due to aluminum acting as a grain refiner and improving the graphite nucleation tendency.
- It has been found that the microstructure normally consists of nodular graphite in a ferritic-pearlitic matrix, and the aluminum addition decreased the extent of bulls-eye structure to give a higher volume fraction of pearlite in the matrix.
- The TEM micrographs from nodule crosssections indicated that the structure of the graphite spherulite is built up by the aggregation of graphite platelets and interplatelet regions.
- Particles can be observed within the spherulite and are compounds containing magnesium, aluminum, silicon and iron.
- Some particles are found at the edge and interface of graphite and matrix. These particles were distributed uniformly all around the graphite nodules. They may have been entrapped during growth and may change the morphology and size of the graphite platelets.

#### 6. ACKNOWLEDGMENTS

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