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# Solidification of gray cast iron

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## Abstract

This article investigates the solidification of hypo, eutectic and hypereutectic gray cast irons, using novel techniques developed by the authors. The nature of the revealed macro and microstructure suggests that the solidification mechanism is different from that usually accepted.

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# 1. Introduction

Several authors have studied the solidification of eutectic gray cast iron of flake graphite morphology (GI) [1–7]. Commonly, the eutectic solidification unit is represented by a nearly spherical shape of austenite and graphite, as shown in Fig. 1. There is general agreement to consider that austenite and graphite grow cooperatively, being both in contact with the liquid phase. This picture of the solidification of GI is supported by the morphology of the graphite flakes, that resemble a rosette, as shown in Fig. 2, and by the fact that the inclusions, generally associated to the microsegregation at the last to freeze melt, are located between such units.

Stead's reagent reveals the microsegregation of phosphorus in GI, and it can delineate the units schematically represented in Fig. 1 in high P irons, usually referred to as "eutectic cells" [8]. Since austenite dendrites are not readily discernible, except in gray irons containing types "D" and "E" graphite, their formation and growth characteristics in GI have received limited attention. Some researchers have considered the role of austenite dendrites in the solidification of GI [2,6,7]. There is no doubt that austenite of hypoeutectic GI grows dendritically. On the other hand, most of the literature work state that austenite can grow with other

morphologies when the carbon content reaches or exceeds the eutectic [3,7].

During the last years the authors of the present article carried out investigations that challenged the validity of the more firmly established models of the solidification of ductile iron (DI) [9–11]. The use of a specially developed technique, that allows to reveal the solidification macrostructure of DI, combined with the use of color metallography techniques that reveal the microsegregation pattern, showed that the macrostructure of DI is formed by relatively large austenite grains, that contain a very large numbers of graphite nodules. This was the case for hypoeutectic, eutectic, and also hypereutectic DI.

The objective of this study is to investigate the solidification mechanism of GI by using the micro and macroscopic techniques, successfully applied for DI in earlier studies.

## 2. Experimental methods

## 2.1. Materials

The melts utilized in the present study were produced by using a 50 kg medium frequency induction melting furnace. Low manganese pig iron, steel scrap and ferroalloys were used as raw materials. Melts were cast in resin bonded sand moulds to produce round bars of 20, 30 and 46 mm diameter. Table 1 lists the chemical composition of the alloys used. The melts were alloyed

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Fig. 1. Schematic illustration of the solidification unit of eutectic GI.



Fig. 2. Morphology of graphite flakes of eutectic GI.

with Cu and Ni in order to provide enough austemperability to carry out the DAAS macrography technique, which is described below.

#### 2.2. Micrographic technique

The color etching technique reveals the solidification microstructure through the use of a reagent that brings up the microsegregation patterns generated during solidification [12]. The etching reagent is made of 10 g NaOH, 40 g KOH, 10 g picric acid and 50 ml distilled water. It must be prepared and handled with great care, since it is caustic and toxic. Etching is carried out at 120 °C (278 °F) for about 2 min.

#### 2.3. Macrographic technique

In order to allow the observation of the macrostructure of DI it is necessary to carry out a thermal process called DAAS (Direct Austempering After Solidification) [9]. In this process, the cast parts are shaken out from the mould short after casting, when their temperature is approximately 950 °C, and transferred to a furnace held at 900 °C, where they remain for 30 min to allow temperature stabilization. The parts are then austempered in a molten salt bath held at 360 °C, for 90 min. A relatively high austempering temperature is used to obtain a high amount of retained austenite after treatment. After the DAAS treatment, the matrix microstructure shows a fine mixture of ferrite needles and retained austenite. A schematic of the thermal cycle is shown in Fig. 3a. The DAAS treatment is certainly laborious and cannot be used under usual molding circumstances. Nevertheless, it is the only method capable of revealing low alloy ductile cast iron macrostructures, to the best of the author's knowledge. After this procedure, the retained austenite keeps the crystalline orientation defined during solidification. Therefore, etching with Picral (5%) reveals the grain structure of the solidification austenite. It must be emphasized that such macrostructure will be not visible after a conventional austempering treatment, as that shown schematically in Fig. 3b, since the microstructure will be formed starting from a fine grain recrystallized austenite structure obtained after austenitizing. This technique has not been applied before to GI. As a first approach it will be used on GI following the same procedure developed for DI.



Fig. 3. (a) DAAS thermal cycle, (b) regular austempering thermal cycle.

Table 1	
Chemical	composition

Melt	Chemical composition [wt.%]								
	CE	С	Si	Mn	Cu	Ni			
Hypoeutectic	3.94	2.99	2.84	0.23	0.96	0.46			
Eutectic	4.27	3.28	2.95	0.22	0.93	0.65			
Hypereutectic	4.64	3.61	3.11	0.18	1.05	0.68			

CE: carbon equivalent.

# 3. Results and discussion

Fig. 4a–c show the unetched microstructure of the 30 mm diameter samples of hypoeutectic, eutectic, and hypereutectic melts, respectively. Predominately type A lamellar graphite structure is observed in all cases. Fig. 5a–c show the macrostructure revealed by the DAAS technique on the same samples of Fig. 4. In all cases, a relatively large grained macrostructure is observed on the sample surface, including the hypereutectic alloy. This is, to the best of our knowledge, the first time such structure is revealed on sand cast GI samples solidified normally. The grains, or solidification units, are much larger than expected for the solidification model based on nearly spherical units (eutectic cells) represented in Fig. 1.

As it was mentioned before, the eutectic cells are usually revealed through the microsegregation of phosphorus by the application of the Stead reagent. Nevertheless, it is important to point out that this method



Fig. 4. Microstructure of unetched samples of 30 mm round bars. (a) Hypoeutectic melt, (b) eutectic melt, (c) hypereutectic melt.



Fig. 5. Macrostructure of 30 mm diameter round bars. (a) Hypoeutectic melt, (b) eutectic melt, (c) hypereutectic melt.

does not prove that areas separated by microsegregation of phosphorus are in fact different grains, because it does not etch differentially grains, or volumes, of different crystal orientation.

It is remarkable that the grain structure has similar size among samples of the same diameter, regardless its carbon equivalent. The only difference is that the hypoeutectic GI shows a more pronounced columnar structure. Fig. 6 shows the macrostructure of the samples of 20, 30 and 46 mm diameter of the hypereutectic melt. Note that the grain size slightly increases when the diameter increases.

The presence of such large grains indicates that large portions of the sample have the same austenite crystalline orientation. It should then be possible to find indications of austenite growth inside a solidification grain. This was investigated by tracing the microsegregation inside each grain by using the color metallography technique. The results show the presence of austenite dendrites for GI of all carbon equivalent values investigated. As an example, Fig. 7 shows the solidification microstructure of the hypereutectic melt. It is remarkable that the graphite colonies do not show an interdendritic morphology, but are immersed in the dendrite stem in many places, as pointed by the arrows. This explains why such dendrites have not been identified before, through the observation of the distribution of flake graphite. It also suggests that flake graphite may grow to some extent, most probably during



Fig. 6. Macrostructure of round bars of hypereutectic melt. (a) 20 mm, (b) 30 mm, (c) 46 mm.

the last stages of solidification or during solid state graphitization, not in contact with the melt but by C diffusion through an austenite envelope.

The presence of such large grains and the morphology of microsegregation patterns suggest that the so-called eutectic cells are not actual individual solidification units, but large groups of them have a common origin in a very large austenite dendrite.

The observations lead to propose the following explanation of GI solidification. Very thin or skinny austenite dendrites nucleate and grow to large extent at, or below, the temperatures pointed on Fig. 8. Any graphite particles that may be present in the melt at this stage are engulfed by this dendritic array. This is most probably effectively taking place in the case of hypereutectic GI. Existing particles at the intradendritic melt, or newly nucleated graphite nuclei at the supersaturated intradendritic melt, make contact with austenite branches and begin cooperative growth, forming the units called eutectic cells. It is possible to speculate that, for both hypereutectic and hypoeutectic GI, there is not a nucleation event for eutectic cells, but the solidification process is dominated by the initial growth of relatively large austenite dendrites that provide a large density of austenite seeds on which the formerly called eutectic



Fig. 7. Black and white print of hypereutectic melt after color etching.



Fig. 8. Schematic section of the eutectic region of the Fe-C equilibrium diagram.

cells can form. These units are not solidification grains, as it is commonly accepted. A great number of them are present into each grain.

The proposed mechanism is supported by observations of other authors. Ruff and Wallace [2] point out that austenite dendrites are present in gray iron of hypo, hyper and eutectic composition, and that the nucleation of eutectic austenite takes place "on and near the primary austenite dendrites". Diószegi et al. [7] state that for hypoeutectic alloys "the place for the eutectic nucleation is believed to be close to the interface of primary austenite in the segregated liquid". These studies do not state that there is no new nucleation of austenite, but they suggest that there is a link between austenite dendrites and the nucleation of eutectic austenite.

Numerous experiments have demonstrated that the number of eutectic cells per unit area is increased by the addition of inoculants to the melt. The solidification model proposed in this work does not deny this mechanism. It is clear that the inoculation process increases the nucleation rate of graphite, then a larger number of graphite nuclei would cause a more frequent interaction between austenite branches and graphite, leading to a larger number of smaller units of coupled eutectic cells. As it is well known the observations of the eutectic cells is very useful to relate solidification structure characteristics with properties of the cast part.

## 4. Conclusions

• The DAAS macrographic technique has been successfully applied to reveal the macrostructure of hypo, hyper and eutectic flake gray cast irons.

- The macrostructure of sand cast hypo, hyper and eutectic GI show relatively large grains in all cases.
- Color metallography techniques were used to reveal the austenite dendrites locations and its interaction with graphite flakes. Graphite flakes frequently cross austenite dendrite stems, suggesting that such flakes can continue growing after they have been enveloped by austenite.
- This study proves that austenite dendrite growth is predominant not only for hypoeutectic but also for hypereutectic gray irons.
- The units usually called "eutectic cells" are not solidification grains, as it is commonly accepted. A great number of them are present into each grain.

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