Preparation of Cast Iron Foundry Alloys

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Cast irons can exhibit a wide range of microstructural constituents depending upon their composition and heat treatment. Their preparation is difficult due to the need to properly retain the graphite phase when it is present. The presence of micro-shrinkage cavities also presents problems, particularly controlling bleedout of fluids and etchants. Metallographic examination may involve only qualitative assessments, for example, to define the type and relative size of the graphite phase and identify phases and other constituents such as nitrides and inclusions. There has been a trend in the foundry industry to become more quantitative with image analysis measurements of the amounts of phases, graphite shapes, and nodule density. In our research work, we quantify structures fully. However, most foundries do not need to be as rigorous in quality control studies.

Preparation Procedures

For our work, which often involves quantitative measurements using image analysis, we must faithfully reveal the true structure. Thus, our preparation procedure is more elaborate than that used by many foundries. We utilize color tint-etching methods extensively, and these require a very high-quality preparation practice.

For the work shown here, I used the following procedure with slight modifications depending upon whether or not the specimen was to be etched and if a tint etch was to be used. Mounted specimens were placed in a holder for six (usually) specimens. Central force and complementary rotation (platen and holder rotating in same direction) were used. The steps were:

1) 120 grit SiC paper, 300 rpm, 100N (22.5 Ibs.) force until all surfaces are coplanar (water coolant)
2) 240 grit SIC paper, 300 rpm, 100N force for 2 minutes (water coolant)
3) 9µm METADI® Diamond Paste on an ULTRA-PAD™ Cloth, 150 rpm, 100N force for 3 minutes
4) 3µm METADI® Diamond Paste on a TEXMET® 1000 pad, 150 rpm, 120N (27 Ibs) force, for 3 minutes
5) 1µm METADI® Diamond Paste on a TEXMET® 1000 pad, 150 rpm, 100N force, for 2 minutes
6) Specimens etched lightly with 4% HNO, in alcohol (nital)
7) MASTERPREP™ Alumina Polishing Suspension used on a MICROCLOTH® Polishing Cloth wet with water, 150 rpm, 90-120N (20-27 Ibs) force (depending on degree of etch), for 1.5-2 minutes (depending on degree of etch)

(WebMasters' Note: For those unfamiliar with Buehler products; ULTRA-PAD™ is a hard woven polyester cloth, TEXMET® 1000 is a napless nonwoven chemotextile polishing pad, MICROCLOTH® is a medium nap synthetic rayon cloth, and MASTERPREP™ is a finely dispersed non-agglomerated .05 micron Alumina suspension.)

METADI® Fluid was used as the lubricant/extender with the diamond abrasives. After each polishing step (Nos. 3, 4, 5 and 7), the specimens were washed with alcohol and blown dry with compressed air (which must be clean and dry). Washing with water sometimes results in corrosion

stains on the surface, so I usually use only alcohol for the final cleaning step (No. 7). Individual force can also be used, especially if I do not have enough specimens to fill the holder (divide the force values given above by 6 to determine the individual force to use).

Microstructures

One should always begin microstructural investigations by examining the as-polished specimen before etching. This is a necessity, of course, for cast iron specimens if we are to properly examine the graphite phase. Brightfield vertical illumination will be our starting point, but the benefits of crossed polarized light will also be explored.

Cast irons with a composition equivalent to about 4.3% C solidify as a eutectic. Because cast irons are not simple binary Fe-C alloys, it is usual practice to calculate the carbon equivalent (CE) value which is the total carbon content plus one-third the sum of the silicon and phosphorus contents. If the CE is > 4.3, it is hypereutectic; if it is < 4.3, it is hypoeutectic. Table 1 - lists the CE values and compositions for each cast iron shown in this issue.

In the Fe-C system, the carbon may exist as either cementite, Fe,C, or as graphite. So the eutectic reaction is either liquid transforming to austenite and cementite at about 1130°C or liquid transforming to austenite and graphite at about 1135°C. Addition of elements such as silicon promote graphite formation. Slow cooling rates promote graphite formation, while higher rates promote cementite. The eutectic grows in a cellular manner with the cell size varying with cooling rate which influences mechanical properties.

Gray lron

Figure 1 shows interdendritic flake graphite in a hypoeutectic alloy (see Table 1 for composition of alloys) where proeutectic austenite forms before the eutectic reaction. This type of graphite has been given many names. In the US it is referred to as Type D (ASTM A247) or as undercooled graphite. It was thought that the fine size of the graphite might be useful, but it is not technically useful as it always freezes last into a weak interdendritic network.

Figure 2 shows more regularly-shaped graphite flakes in an alloy (Table 1) of higher carbon content, although still hypoeutectic. While flake lengths in Figure 1 are roughly 15-30µm, flake lengths in Figure 2 are in the 60-120µm range. Figure 3 shows somewhat coarser flakes (250-500µm length range) in a higher carbon content cast iron (Table 1).
Other graphite forms are also observed. For example, Figure 4 shows disheveled graphite flakes in a casting. Note that a few nodules are present. This appears to be a mix of B- and D-type flakes. Figure 5 shows a hypereutectic gray iron where very coarse flakes form before the eutectic which is very fine. This is similar to C-type graphite.

Nodular Iron

The addition of magnesium ('inoculation') desulfurizes the iron and causes the graphite to grow as nodules rather than flakes. Moreover, mechanical properties are greatly improved over gray iron; hence, nodular iron is widely known as'ductile iron'.
Nodule size and shape perfection can vary depending upon composition and cooling rate. Figure 6 shows fine nodules, about 15-30µm in diameter, while Figure 7 shows coarser nodules (about 30-60µm diameter) in two ductile iron casts (see Table 1 for compositions). Note that the number of nodules per unit area is much different, about 350 per mm^2 vs.125 per mm^2, respectively.

Compacted Graphite

Compacted graphite is a more recent development made in an effort to improve the mechanical properties of flake gray iron. Figure 8 shows an example where the longest flakes are in the 60-120µm length range. Compare these flakes to those shown in Figures 2 and 3.
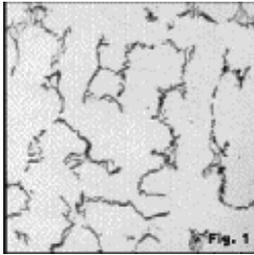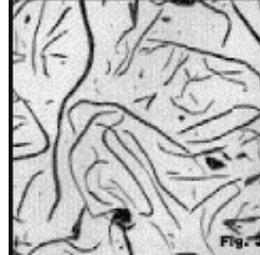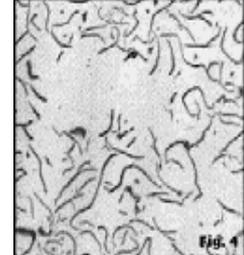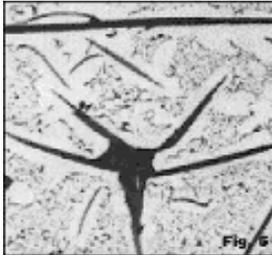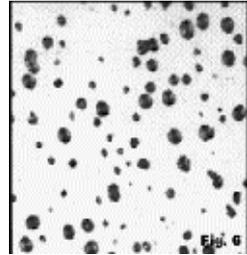

Figure 1

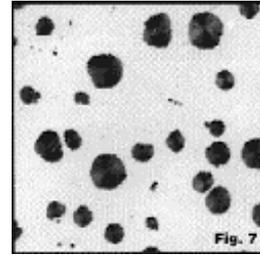
Figure 2
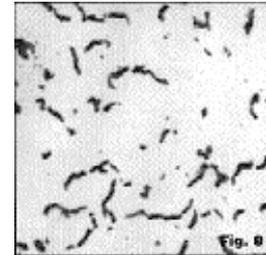

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

Crossed-Polarized Light

Examination of graphite in crossed polarized light requires a very well-prepared specimen; otherwise, the matrix phase exhibits a heavy scratch pattern, and the graphite growth pattern will not be visible. Minor scratches can be tolerated in routine work.

Etching

To see details of the matrix microstructure, specimens must be etched. The Polish standard for cast irons (PN-61/H-0503) recommends three etchants. The first is 4% alcoholic nitric acid ("nital") used at room temperature to reveal the ferrite grain boundaries and reveal phases and constituents such as cementite and pearlite. The second is alkaline sodium picrate (25g NaOH, 2g picric acid and 75 mL distilled water) used at 60 to 100°C for up to 30 minutes (1-3 minutes is usually adequate). This is used to color cementite yellow to brown (ferrite is not colored). The third is the standard version of Murakami's reagent (10g KOH, 10g potassium ferricyanide and 100 mL distilled water) used at 50°C for 3 minutes to color iron phosphide dark yellow or brown (cementite and ferrite not colored).

500x

500x

800x

250x
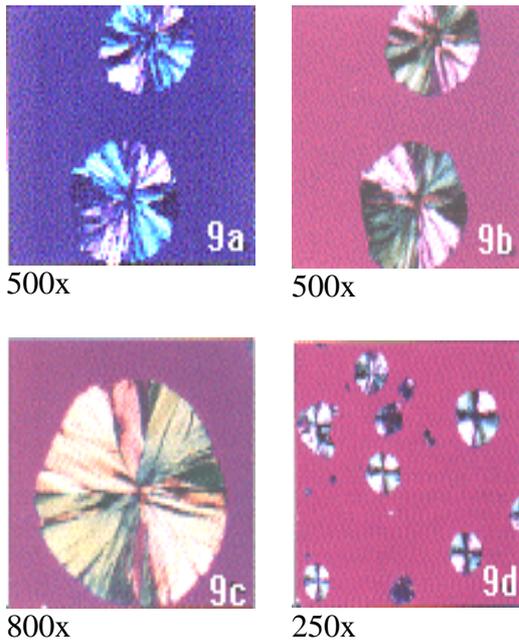
The polarizer and analyzer filters are placed in the crossed position (i.e., 90° to each other which produces the darkest matrix appearance), and a sensitive tint plate (lambda plate) may be inserted to further enhance coloration.The lambda plate may be adjustable on some microscopes which changes the color pattern, as illustrated in Figures 9a and b. Personally, I prefer to set the background color to magenta, as in Figure 9b. Working at a very high magnification, Figure 9c, provides more structural detail than at low magnification, Figure 9d, but requires high quality, stress-free objectives. Note the classic cross pattern seen at low magnification, Figure 9d.

Flake graphite can also be studied using polarized light, such as illustrated in Figure 10. The color varies with the crystallographic orientation of the graphite, and some internal details of the flake structure are better revealed in color.


Fig.10
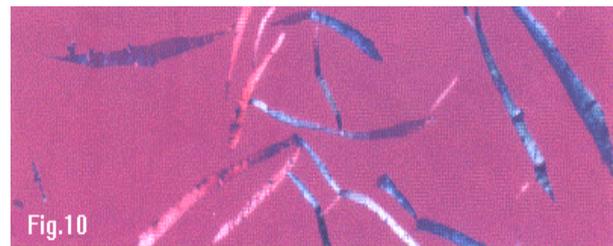
Gray Iron

As an example, Figure 11 shows a flake graphite specimen (see Table 1) etched with 4% nital. The matrix is predominantly pearlitic (colored tan, blue, and brown) and shows patches (arrow) of the ternary eutectic (ferrite, cementite, and phosphide).

Fig.11
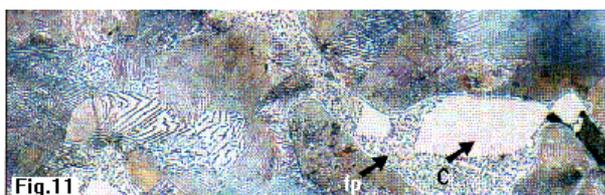
Microstructure of gray iron containing a pearlitic matrix and the ternary eutectic (4% nital, 250x). The larger white particles (arrow-C) are cementite while the adjacent area (arrow-fp) contains ferrite and iron phosphide.

In comparison, Figure 12 shows the matrix structure of the specimen shown in Figure 2. The ternary phosphide is not present. The matrix is all fine pearlite.
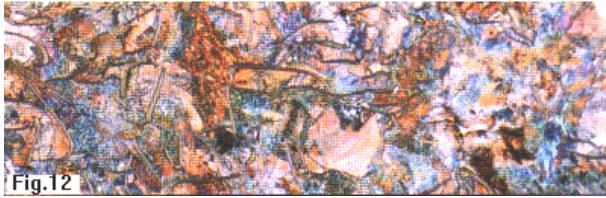
Pearlitic matrix of the gray iron specimen shown in Fig. 2 etched with 4% nital, 100x.

Besides the ternary ferrite-cementite-iron phosphide eutectic and the previously mentioned binary eutectics (austenite and cementite and austenite and graphite), it is possible to obtain a binary ferrite-iron phosphide eutectic in cast iron. Figure 13 shows the binary ferrite-iron phosphide psuedo- eutectic in the specimen previously shown in Figure 10, after etching with hot Murakami's reagent which colors the phosphide brown but does not color ferrite.

Gray iron specimen containing a binary ferrite-iron phosphide eutectic with the phosphide colored by the hot Murakami's etch, 100x.

Nodular Iron

Ductile iron specimens can have a wide range of matrix structures depending upon composition and as-cast cooling rate. Figure 14 shows a fully ferritic matrix after etching with 4% nital. Figure 15 shows a specimen with a pearlitic matrix and ferrite surrounding the nodules, while Figure 16 shows an example where a very small amount of ferrite remains around each nodule.
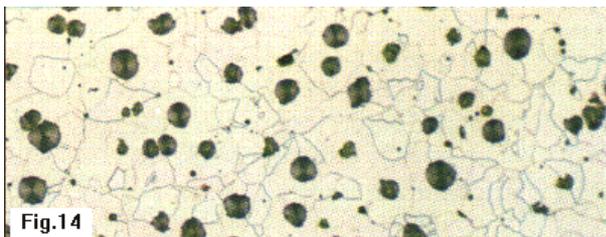

Figure 14. Fully ferritic matrix (note ferrite grain boundaries) of a ductile iron specimen etched with 4% nital, 100x.
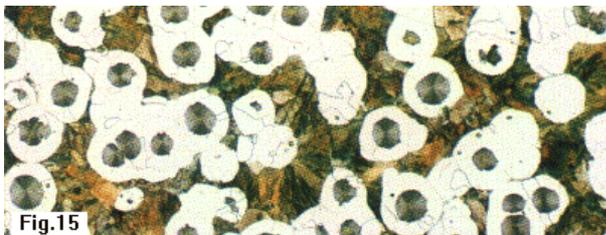

Figure15. Ductile iron specimen with substantial amounts of ferrite and pearlite etched with 4% nital, 100x.
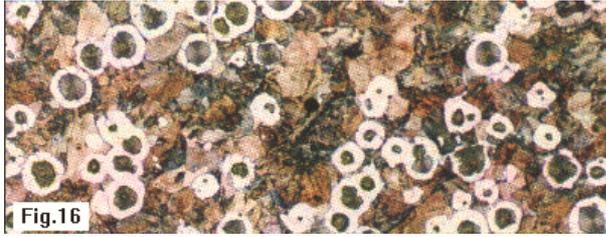
Figure 16. Ductile iron specimen with a pearlitic matrix and a small amount of ferrite surrounding the nodules etched with 4% nital, 100x.

White Cast Iron

The microstructure of white cast iron is best observed after etching. Figure 17 shows a typical example after etching with 4% nital. Note the interdendritic cementite (white) which sometimes has a Widmanstiitten ('spiky') appearance. Austenite formed as the proeutectic constituent before the eutectic reaction (liquid transforms to austenite and cementite) and later transforms to pearlite and cementite upon cooling below the eutectoid temperature, about 723°C.
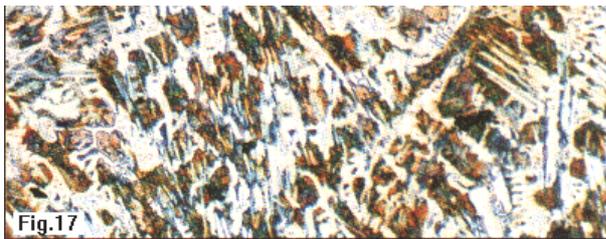

Figure 17. Microstructure of white cast iron containing massive cementite (white) and pearlite etched with 4% nital, 100x.

Figure 18a shows a higher magnification view of this specimen etched with 4% nital. The massive cementite particles are clearly visible appearing to be outlined by the etch. Actually, the cementite is not attacked by the etch, while the surrounding structure is. The 'outline' around the cementite particles is due to light being scattered from the height difference or 'step' around the particles. Note that ferrite surrounds each cementite particle due to local decarburization. The pearlite is colored.
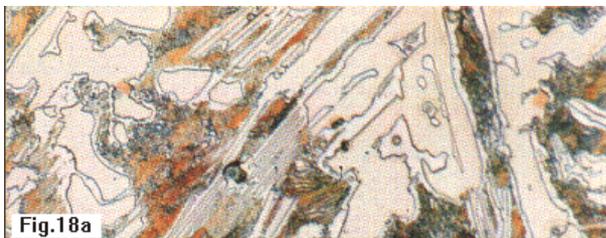

Figure 18a. High magnification view (400x) of the white cast iron specimen shown in Figure 17, etched with 4% nital.

Figure 18b shows the effect of etching this specimen with alkaline sodium picrate which colors the massive cementite brown. The cementite in the pearlite constituent is colored tan and blue. Ferrite is not colored.

Figure 18b. High magnification view (400x) of the white cast iron specimen shown in Figure 17, etched with alkaline sodium picrate.

Conclusions

Modern polishing materials and procedures can be employed very effectively to reveal the microstructure of cast iron specimens. Graphite retention, always a problem with these metals, can be accomplished with a minimum of difficulty. Crossed polarized light is very useful for observing graphite substructure. Etching brings out the matrix constituents.

Selective etching with color producing films, briefly discussed here, is a highly informative tool. This will be discussed in more detail in a future issue.

Table 1: Composition of Cast Irons (Wt. %, balance Fe)

Figure #	C	Si	Mn	P	S	Ni	Mg	Cu	Other	CE
1	2.1	2.8	0.38	0.06	0.03	-	-	-		3.05
2, 12	2.8	1.85	1.05	0.04	0.025	-	-	-		3.43
3	3.5	2.95	0.40	0.08	0.02	0.13	-	0.15		4.51
4	3.5	2.7	0.55	0.13	0.02	-	0.03	-	0.46 Al	4.44
5	4.3	1.5	0.5	0.12	0.08	-	-	-		4.84
6	3.45	2.55	0.30	0.04	0.01	0.80	0.07	0.55		4.31
7	3.6	2.9	0.14	0.04	0.02	0.16	0.06	-		4.58
8	3.7	2.3	0.21	0.03	0.01	0.82	0.02	-		4.48
9	3.6	2.9	0.14	0.04	0.02	0.16	0.06	-		4.58
10, 13	3.3	1.64	0.31	1.42	0.11	-	-	-		4.32
11	3.3	1.9	0.3	0.49	0.04	-	-	1.2		4.10
14	3.7	1.25	0.03	0.02	0.02	0.24	0.06	-		4.12
15	3.7	1.25	0.2	0.02	0.02	0.22	0.06	-		4.12
16	2.8	2.8	0.2	0.045	0.02	0.88	0.04	0.48	-	3.75
17, 18	3.0	2.7	0.45	0.07	0.025	-	-	-	Te to mould	3.92