Metallurgical Engineering II – Laboratory Manual

Metallographic Specimen Preparation, Types of Etchants and Optical Microscopy of Cast Irons

Group: ..... | **Date:** .....

1. Objective

The aim of this experiment is for students to learn the complete metallographic preparation chain (sectioning, mounting, grinding, polishing, etching) through hands-on practice, to understand the mechanisms and application areas of commonly used etchants, and to identify the microstructures of the cast iron types widely used in industry (gray, ductile, malleable, white) with a metallurgical optical microscope. At the end of the experiment, the student should be able to:

- Prepare a defect-free metallographic specimen,
- Select a suitable etchant for a given material and justify the choice,
- Determine the graphite type (A, B, C, D, E) and size in gray cast iron,
- Interpret the nodule shape and matrix structure (ferrite/pearlite) of ductile iron,
- Relate microstructure to mechanical properties.

2. Theoretical Background

2.1 Importance of Metallography

Metallography is the examination of the internal structure of a material (phases, grains, inclusions, graphite, carbides, etc.) under magnification. Microstructure is the direct result of chemical composition, solidification and cooling conditions, and heat treatment history. It is therefore one of the most fundamental tools in quality control, failure analysis and process development. A reliable result can only be obtained when **the specimen is prepared without altering the true structure**: scratches, smearing, deformed layers or graphite pull-out created during preparation can produce “structures” that do not exist in reality.

2.2 Sampling and Sectioning

- The specimen must be taken from a location representative of the region to be examined (for castings, section thickness, distance from the surface and segregation are considered).
- Sectioning must be done with an abrasive cutting wheel using **plenty of coolant**. Excessive heating changes the microstructure (e.g., tempering effects in white cast iron).
- Typical specimen size: Ø 25 – 30 mm or 20 × 20 mm, height 10 – 15 mm.

2.3 Mounting

Small, irregular specimens or those requiring edge examination are mounted in resin for easier handling and **edge retention**.

Method	Material	Characteristics
Hot mounting	Bakelite (phenolic), acrylic	Fast, economical; requires ~150 °C and pressure

Method	Material	Characteristics
Cold mounting	Epoxy, acrylic resin	For heat-sensitive or porous specimens; long curing time
Mechanical clamping	Clips, holders	For large and flat specimens

2.4 Grinding

The purpose of grinding is to **progressively** remove cutting marks and the deformed layer. Silicon carbide (SiC) waterproof papers are used, and the specimen is ground **under running water**.

Basic rules

1. Proceed from coarse to fine grit: **P120 → P240 → P400 → P600 → P800 → P1200** (P2500 if needed).
2. At each step, grind until the scratches from the previous step are completely removed.
3. Rotate the specimen by **90°** at each step change; this removes old scratches in the perpendicular direction and makes it easy to check completion.
4. Apply moderate, constant pressure; excessive pressure creates a deep deformation zone.
5. Wash the specimen and hands between steps; otherwise coarse grit is carried onto the finer paper and causes deep scratches.

2.5 Polishing

Polishing removes grinding marks to produce a mirror-like surface. Diamond suspensions or oxide powders (alumina, colloidal silica) are used on cloths.

Stage	Abrasive	Cloth	Time (approx.)
Rough polishing	6 µm or 3 µm diamond	Low-nap / woven cloth	2 – 4 min
Intermediate polishing	1 µm diamond	Medium-nap cloth	1 – 3 min
Final polishing	0.05 µm alumina or 0.04 µm colloidal silica	Napped cloth	30 s – 2 min

Special warnings for cast irons: Graphite is soft and brittle; long polishing times, high pressure or a hard napped cloth cause **graphite pull-out**, deformed nodules and comet tails. Therefore keep polishing times short, apply light pressure and prefer low-nap cloths. Otherwise the nodularity or graphite content will appear different from the true value.

After polishing, rinse the specimen under running water, rinse with ethanol and dry with warm air. A well-prepared specimen should appear free of scratches and stains under the microscope before etching.

2.6 Etching and Types of Etchants

On a polished surface, phases and grain boundaries reflect light in the same way, so there is no contrast (only graphite, inclusions and cracks are visible). **Etching** chemically attacks the surface in a controlled manner, creating height or color differences between phases and grain boundaries, which provides contrast.

Etching methods

- **Chemical etching:** Immersion or swabbing (the most common method).
- **Electrolytic etching:** Anodic dissolution by an applied current (often used for stainless steel, Al and Cu alloys).

- **Heat tinting:** Phase-dependent coloring through controlled oxidation.
- **Color (tint) etching:** Reagents such as Beraha and Klemm; phase coloring via thin film formation.
- **Ion / plasma etching:** Surface removal in vacuum (special applications).

Commonly used chemical etchants

Etchant	Composition (approx.)	Application	Time / note
Nital	2 – 5% HNO_3 + ethanol	Steel, cast iron; ferrite grain boundaries, pearlite, cementite	5 – 30 s; most common for cast iron
Picral	4% picric acid + ethanol	Pearlite, cementite, carbide detail in steel and cast iron	10 – 60 s; does not reveal ferrite grain boundaries well
Vilella	1 g picric acid + 5 mL HCl + 100 mL ethanol	Martensitic steels, tool steels, ledeburite	For highly alloyed steels
Kalling No. 2	5 g $CuCl_2$ + 100 mL HCl + 100 mL ethanol	Austenitic/duplex stainless steels	A few seconds – minutes
Murakami	Alkaline $K_3Fe(CN)_6$ solution	Coloring of carbides and cementite; sigma phase	Hot/cold use; distinguishes phosphorus eutectic (steadite)
Alkaline sodium picrate	Picric acid + aqueous NaOH solution	Cementite vs. ferrite separation, white cast iron	Cementite darkens
Stead's reagent	$CuCl_2$ / $MgCl_2$ / HCl / ethanol based	Phosphorus segregation in steel and cast iron	Applied by the instructor
Keller's	HNO_3 + HCl + HF + water	Al alloys	Contains HF, instructor supervision only
$FeCl_3$ solution	$FeCl_3$ + HCl + water	Cu and its alloys	Swabbing or immersion

Safety note: In this experiment students work only with **2% Nital** and (if needed) **4% Picral**. Dry picric acid can be **explosive**; reagents containing HF or concentrated acids are used only under instructor supervision.

Effect of etching time: Under-etching = low contrast; over-etching = loss of detail, darkening, surface stains. If etching is faulty, the specimen is re-polished (final stage) and etched again.

2.7 Metallurgical (Reflected-Light) Optical Microscope

A metallurgical microscope examines opaque specimens using **reflected light**. Light is sent through the objective onto the specimen surface and collected back through the same objective.

- **Total magnification = objective magnification × eyepiece magnification** (e.g., 50× objective × 10× eyepiece = 500×).
- Typical objectives: 5×, 10×, 20×, 50×, 100×.
- **Illumination modes:** Bright field (most common), dark field (cracks, pores, inclusions), polarized light (anisotropic phases, graphite), DIC (surface relief).
- The specimen surface must be **perpendicular** to the microscope axis; a specimen leveling press and plasticine are used for this.
- Every image must include a **scale bar** and magnification information.

Operating steps: (1) Place the specimen on the stage, (2) focus with a low-magnification objective (5× – 10×), (3) locate the region of interest, (4) move gradually to higher magnification, (5) adjust the aperture diaphragm and brightness, (6) record the image.

2.8 Cast Irons and Microstructure Classification

Cast irons are Fe–C–Si alloys containing approximately **2.0 – 4.5% C** and **1 – 3% Si**. Depending on solidification and cooling conditions, carbon exists either as **free graphite** or as **cementite (Fe_3C)**. The microstructure is described by two components: (i) the **shape, distribution and size of graphite**, (ii) the **metallic matrix** (ferrite, pearlite, ferrite + pearlite, bainite, austenite, martensite).

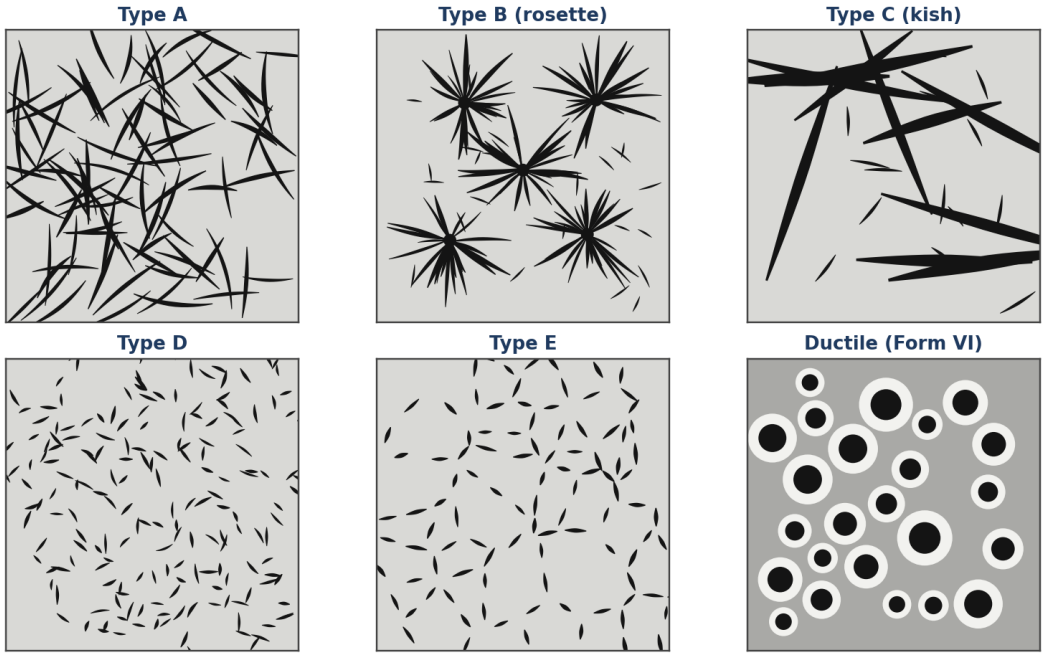
Type	Graphite / carbide shape	Typical matrix	Properties and applications
Gray cast iron	Lamellar (flake) graphite	Pearlite, ferrite + pearlite	Good castability, vibration damping, machinability; engine blocks, discs, heads, machine tool beds
Ductile (nodular) cast iron	Spheroidal graphite (via Mg or Ce)	Ferrite, pearlite, ferrite + pearlite, ADI	High strength and ductility; crankshafts, pipes, gears, suspension parts
Compacted graphite (CGI)	Worm-like graphite with blunt ends	Pearlite + ferrite	Properties between gray and ductile; engine blocks, exhaust manifolds
Malleable cast iron	Temper carbon (irregular rosettes / clusters)	Ferrite or pearlite	Obtained by annealing white cast iron; thin-section, ductile parts
White cast iron	No graphite; cementite, ledeburite	Pearlite + cementite (ledeburite)	Very hard, wear-resistant; grinding balls, liners

Graphite Types in Gray Cast Iron (ASTM A247 / ISO 945)

Graphite flakes in gray cast iron are classified by their distribution into types **A, B, C, D and E**. Classification is made on the **unetched (polished)** specimen at 100× magnification.

Type	Appearance	Formation condition	Comment
A	Uniformly distributed, randomly oriented, medium-length flakes	Moderate cooling rate, near-eutectic composition, good inoculation	**The most desired type in industry**; good strength and machinability
B	**Rosette** pattern: fine at the center, longer flakes outward	Slightly higher cooling rate, near-eutectic composition	Seen near the surface and in thin sections; somewhat higher strength than A
C	**Kish graphite**: thick, coarse, straight plates	Hypereutectic composition (high C and Si)	Low strength; high thermal conductivity
D	**Interdendritic, fine, randomly oriented** graphite	Rapid cooling, undercooling	May accompany a ferritic matrix; seen in thin sections
E	**Interdendritic, preferentially oriented** graphite	Hypoeutectic composition, rapid cooling	Wear and non-uniform mechanical properties; similar to D

Graphite size (ASTM A247): Classified from **1 (coarsest) to 8 (finest)** at 100× according to the longest flake length; as size decreases, strength increases and thermal conductivity decreases.



Unetched specimen, ~100× (schematic drawing)

Figure 1. Schematic drawings of graphite types A – E in gray cast iron and nodular graphite (Form VI) in ductile iron. For real microstructures, use the micrographs you capture in the laboratory and the ASTM A247 reference charts.

Evaluation of Ductile Iron

- **Nodule shape (ISO 945: Form I – VI):** Form VI is ideal spheroidal, Form V imperfect spheroidal, Form III vermicular, Form I lamellar.
- **Nodularity (%):** Ratio of spheroidal nodules to the total graphite count; high-quality ductile iron generally requires **above 80 – 90%**.
- **Nodule count (number/mm²) and size** (ASTM A247 size 1–8).
- **Matrix:** In a Nital-etched specimen, **ferrite** appears white-light and **pearlite** appears dark; the **ferrite ring** around a nodule is known as the “bull’s-eye” structure.

White and Malleable Cast Iron

- **White cast iron:** After Nital etching, **pearlite dendrites** (dark) and the **ledeburite** between them (cementite + pearlite, light) are visible; cementite remains bright white.
- **Malleable cast iron: Temper carbon**, formed by the decomposition of cementite when white cast iron is annealed at 900 – 950 °C, appears as irregular clusters.

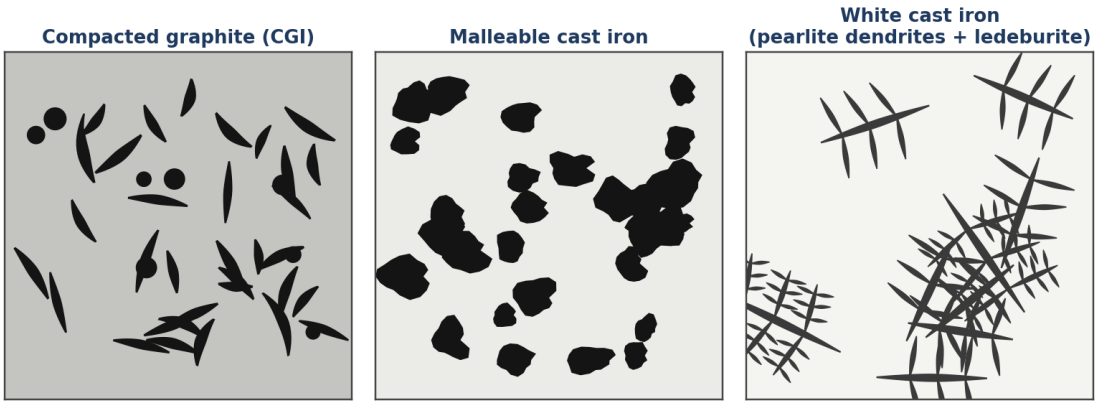


Figure 2. Schematic microstructures of compacted graphite, malleable and white cast iron (white cast iron shown in the etched condition).

3. Materials and Equipment

Specimens: Gray cast iron (type A dominant), gray cast iron (containing type B/D/E), ductile cast iron (ferritic or ferritic-pearlitic), malleable cast iron, white cast iron (at least two specimens per group).

Equipment: Abrasive cutting machine, mounting press or cold-mounting molds, grinding-polishing machine, SiC papers (P120 – P1200), polishing cloths, diamond suspensions (6/3/1 µm), final polishing suspension (0.05 µm), 2% Nital, ethanol, distilled water, towels/cotton, dryer (hair dryer), metallurgical optical microscope (5× – 100× objectives), camera and software for image capture.

Personal protective equipment: Lab coat, safety glasses, nitrile gloves. Etchants containing acid and ethanol are used in a **fume hood** and kept away from open flames.

4. Procedure

Step 1 – Specimen preparation: Record the specimen code. If necessary, perform cutting or mounting under the instructor’s supervision. Slightly chamfer the edges.

Step 2 – Grinding: Start with P120 and proceed to P1200 under running water, rotating the specimen 90° at each step. Check scratch direction and cleanliness at every step. *Time: ~1 – 2 min per step.*

Step 3 – Polishing: Rough polish with 3 µm diamond, intermediate polish with 1 µm, and finish with a short final polish using 0.05 µm. Polish with continuous light pressure on the cloth, rotating the specimen counter to the platen direction. For cast iron, keep polishing times **short**. After washing, ethanol rinsing and drying, check the surface under the microscope.

Step 4 – Examination of the unetched specimen: At 100× magnification, examine graphite shapes, size and distribution. In gray cast iron, determine the graphite type (A, B, C, D, E) and size; in ductile iron, estimate nodule shape and nodularity. Image at least **3 different areas**.

Step 5 – Etching: Immerse the specimen in 2% Nital for 5 – 15 s or swab it (Nital is ethanol-based; work in the fume hood). Immediately wash under running water, rinse with ethanol and dry with warm air. The surface should appear matte gray.

Step 6 – Examination of the etched specimen: Examine the matrix at 100×, 200× and 500×: ferrite (light), pearlite (dark, lamellar), cementite/ledeburite (bright white), steadite (if present). Determine the pearlite/ferrite ratio **approximately** or by image analysis / point counting.

Step 7 – Image recording: For each specimen, take **at least 2 images** of the selected structure with a scale bar. Note the etching time and etchant.

5. Data and Observation Table

Specimen code	Type (estimated)	Graphite type / shape	Graphite size (1–8)	Nodularity (%)	Matrix (ferrite / pearlite %)	Etchant and time	Magnification

Sketch / image area: For each specimen, draw or paste the microstructure you observed and indicate the phases with arrows.

6. Common Problems and Solutions

Observation	Possible cause	Solution
Deep, unidirectional scratches	Grinding step skipped or insufficient time	Return to the previous step and remove scratches completely
Comet tails	Hard particle or excessive pressure	Clean cloth, lower pressure
Graphite voids, “torn” graphite	Long polishing, high pressure	Short time, light pressure, low-nap cloth
Stains or water marks on surface	Insufficient washing or drying	Rinse with ethanol and dry quickly
No contrast	Under-etching	Etch again for a short time
Darkened surface, no detail	Over-etching	Re-polish and etch for a shorter time
Rounded edges, focus problems	No edge protection in mounting, over-polishing	Hard cloth, short time; edge-retention resin
Phases that do not exist	Residual deformation layer	Do the earlier steps properly instead of extending final polishing

7. Report Content

The report must include the following sections: (1) Objective, (2) Brief theory (etchant types and cast iron classification), (3) Specimens used and procedure, (4) Microstructure photographs (with scale bar), (5) Observation table, (6) Discussion, (7) Conclusion, (8) References.

8. Questions

1. Why is the specimen rotated by 90° at each step change during grinding?
2. Why is the graphite type in gray cast iron determined before etching, on the polished specimen?

3. What are the application areas of Nital and Picral, and how do they differ? Which one reveals ferrite grain boundaries better?
4. Compare type A, B and E graphite distributions in terms of formation conditions and mechanical properties. Why is type A mostly preferred in industry?
5. List separately the **casting-related** and **specimen-preparation-related** causes that may lead to low nodularity in ductile iron.
6. Which constituents are visible in the microstructure of white cast iron? How can they be distinguished using alkaline sodium picrate or Murakami's reagent?
7. A nodule measured as 25 mm in an image at a total magnification of 500× has what actual diameter in µm?
8. What measures would you take to reduce graphite pull-out during polishing?

9. References

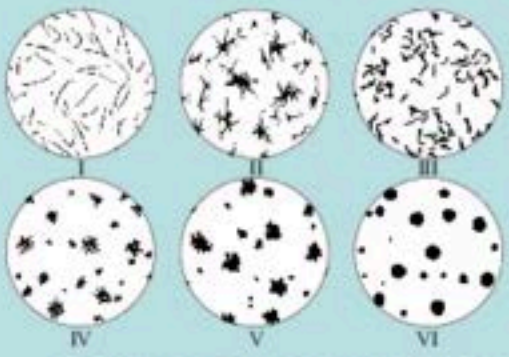
- ASTM A247 – Standard Test Method for Evaluating the Microstructure of Graphite in Iron Castings.
- ISO 945-1 – Microstructure of cast irons – Part 1: Graphite classification by visual analysis.
- ASTM E3 – Standard Guide for Preparation of Metallographic Specimens.
- ASTM E407 – Standard Practice for Microetching Metals and Alloys.
- ASM Handbook, Vol. 9: Metallography and Microstructures.
- Vander Voort, G.F., *Metallography: Principles and Practice*, ASM International.
- Callister, W.D., *Materials Science and Engineering: An Introduction*, Wiley.

GRAPHITE STRUCTURES IN CAST IRONS

Elkem

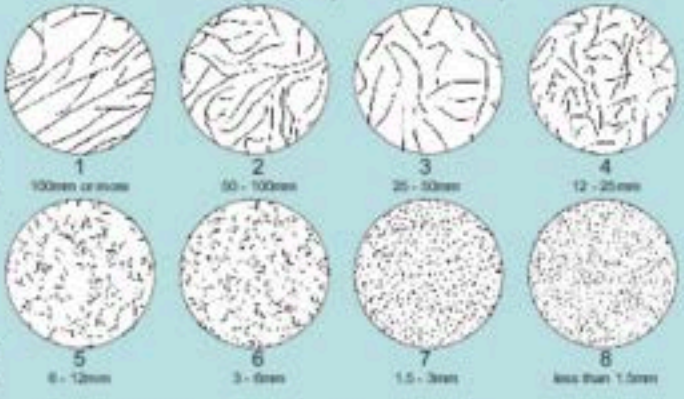
GRAPHITE FORMS

Diagrammatic representations of the standard graphite forms in cast irons



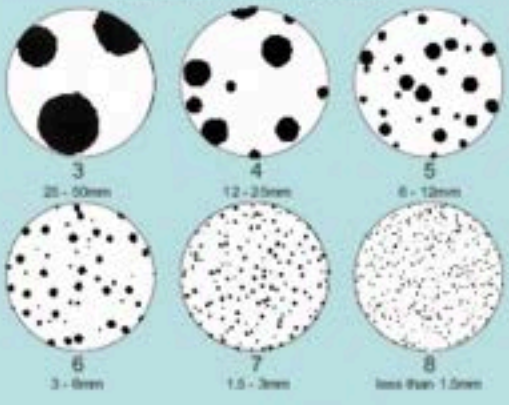
GRAPHITE SIZES IN GREY CAST IRON

Diagrammatic representations of the standard flake graphite sizes in grey cast iron at x100 magnification (Form I Distribution A)



GRAPHITE SIZES IN DUCTILE IRON

Diagrammatic representations of the standard graphite nodule sizes in ductile iron at x100 magnification(Form VI)



INOCULANTS

Elkem manufactures and markets a series of high quality inoculants to treat cast iron and ensure the production of the ideal graphite form, distribution and freedom from chill (carbides).

Inoculant	Active Elements	Advantages and Uses
Superseed® 75/10	Si, Sr	Low Al content. Low eutectic cell count through-cover addition rates. High chill reduction.
Foundrisil®/Barinoc®	Si, Ba, Ca, (Al)	High chill reduction. Resistance to fading. Used in all cast irons.
Ultraseed®	Si, Ca, Ce, (Al,S,O)	High nodule count. Improved nodularity. Helps to prevent micro-shrinkage. Neutralises "dead" iron.
Roseed®	Si, Ca, Ce, (Al)	High nodule count. Improved nodularity. Controls tramp elements in ductile irons. Low S grey iron.

All inoculants are available in sizes suitable for ladle or in-stream additions. Other inoculants for specialist applications are available.

NODULARISERS

Elkem manufactures and markets a range of high quality nodularising alloys to produce ductile and compacted graphite cast irons. They contain balanced ratios of the active elements to control reactivity, minimise slag and neutralise tramp elements.

Product	Active Elements	Advantages and Uses
Elmag®	Si, Mg, Ca, Rare Earths	Magnesium contents from 4 to 10%. Calcium from 0.2% to 3.5%. Rare Earths from 0.1 to 3%. Allows the choice of the ideal alloy for all foundry conditions.
Remag®	Si, Mg, Ca, Rare Earths	High RE, low Mg alloy for low reactivity. Suited to thin section ductile iron castings.
Lumet™	Si, Mg, Ca, La	Alloy for in-the-mould treatment of ductile iron. Helps to prevent shrinkage.
CompactMag™	Si,Mg, Ca, Rare Earths	Highly effective alloy for the production of compacted graphite irons with reduced section sensitivity and wide production window.

Please refer to your local Elkem representative for further information on the range of products available for grey, compacted and ductile irons.

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The graphite form, distribution and size diagrams are taken from the international standard EN ISO 945:1994 by kind permission of the British Standards Institution, London, W4 4AL.

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GRAPHITE DISTRIBUTION IN GREY CAST IRON



TYPE A
Random flake graphite in a uniform distribution





TYPE B
Rosette flake graphite



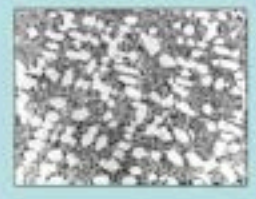


TYPE C
Kish graphite
(hyper-eutectic compositions)





TYPE D
Undercooled flake graphite

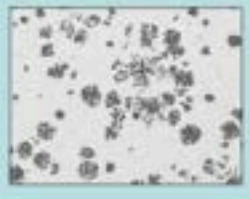




TYPE E
Interdendritic flake graphite
(hypo-eutectic compositions)



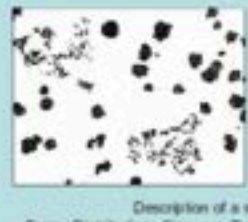
SOME ABNORMAL GRAPHITE FORMS IN DUCTILE IRON



EXPLODED GRAPHITE
Caused by high purity charge materials and excess rare earth additions in high carbon equivalent irons or in thicker section castings



CHUNKY GRAPHITE
Caused by high purity charge materials and excess rare earth additions in large section castings or at high carbon equivalent



SPIKY GRAPHITE
Caused by small amounts of tramp elements, e.g. lead, bismuth, antimony and titanium, in the absence of cerium

Description of a structure should follow the convention
Form : Distribution : Size, e.g. Type I.A.4 for a grey cast iron Type VI.5 for a ductile iron

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