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Standard Guide for Categorization of Microstructural and Microtextural Features Observed in Optical Micrographs of Graphite[1]

This standard is issued under the fixed designation D8075; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers the identification and the assignment of microstructural and microtextural features observed in optical micrographs of graphite. The objective of this guide is to establish a consistent approach to the categorization of such features to aid unambiguous discussion of optical micrographs in the scientific literature. It also provides guidance on specimen preparation and the compilation of micrographs.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D7219 Specification for Isotropic and Near-isotropic Nuclear Graphites

3. Terminology

3.1 The definitions listed below cover terms used in this guide and apply specifically to the optical microscopy of graphite. Properties and features not apparent under the optical microscope are avoided where possible. Definitions may not exactly match those adopted in general scientific usage but should not be at variance. General terms have not been redefined with graphite-specific meanings or optical microscopy-specific meanings. As with the identification of features in micrographs, some definitions have become unclear to differences in usage and this guide provides the basis for a more consistent approach.

3.2 *Definitions:*

3.2.1 *accommodation cracks, n*—(also referred to as Mrozowski-like cracks) cracks and voids formed between basal planes and at domain interfaces throughout the graphite microstructure from thermal contraction of the graphite during carbonization/graphitization (sometimes referred to as calcination cracks), from chemical decomposition of the liquid crystal hydrocarbon precursor in graphite manufacture (also referred to as calcination cracks) and following cooling after graphitization (manufacture). In irradiated graphite, they also comprise cracks arising from anisotropic responses to irradiation.

3.2.2 *agglomerate, n—in manufactured carbon and graphite product technology*, composite particle containing a number of grains.

3.2.3 *binder, n*—substance such as coal tar pitch or petroleum pitch, used to bond the coke or other filler material prior to baking.

3.2.4 *crystallite, n—in manufactured carbon and graphite product technology*, a region of regular crystalline structure having parallel basal planes.

3.2.5 *filler, n—in manufactured carbon and graphite product technology*, particles that comprise the base aggregate in an unbaked green-mix formulation (also referred to as coke particles, grist particles, or filler grains).

3.2.6 *filler-binder phase, n—in manufactured carbon and graphite product technology*, mix of finely ground filler (flour) and binder comprising the matrix in which the filler is bound.

3.2.7 *grain, n—in manufactured carbon and graphite*, particle of filler material (usually coke or graphite) in the starting mix formulation. Also referred to as granular material, filler particle, or aggregate material. The term is also used to describe the general texture of a carbon or graphite body, as in the descriptions listed below:

[1] This guide is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.F0 on Manufactured Carbon and Graphite Products.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.2.7.1 *coarse grained, adj*—containing grains in the starting mix that are substantially greater than 4 mm in size.

3.2.7.2 *medium coarse grained, adj*—containing grains in the starting mix that are generally less than 4 mm in size.

3.2.7.3 *medium grained, adj*—containing grains in the starting mix that are generally less than 2 mm in size.

3.2.7.4 *medium fine grained, adj*—containing grains in the starting mix that are generally less than 1 mm in size.

3.2.7.5 *fine grained, adj*—containing grains in the starting mix that are less than 100 µm in size.

3.2.7.6 *superfine grained, adj*—containing grains in the starting mix that are less than 50 µm in size.

3.2.7.7 *ultrafine grained, adj*—containing grains in the starting mix that are less than 10 µm in size.

3.2.7.8 *microfine grained, adj*—containing grains in the starting mix that are less than 2 µm in size.

3.2.7.9 *Discussion*—All of the above descriptions relate to the generally accepted practice of measuring the sizing fractions with a criterion that 90 % of the grains will pass through the stated sieve screen size in a standard particle sizing test.

3.2.8 *highly oriented region, n*—an area of uniform color under polarized light associated with a relatively crystalline unidirectional (at the observed magnification) orientation.

3.2.9 *isotropic nuclear graphite, n*—graphite in which the isotropy ratio based on the coefficient of thermal expansion (25 °C to 500 °C) is 1.00 to 1.10.

3.2.10 *mesophase, n*—fluid phase (discotic nematic liquid crystal phase) converted to graphite during pyrolysis.

3.2.11 *mosaics, n*—term used to describe texture consisting of a grouping of isochromatic domains, often subdivided by grain size. The following terms may be encountered relating to these microtextural features:

3.2.11.1 *mosaic cluster, n*—an identifiable grouping of similar-sized mosaic texture.

3.2.11.2 *mosaic ribbon, n*—an identifiable ribbon-shaped or strand grouping of mosaic texture.

3.2.11.3 *supra mosaic, n*—aligned region of coarse mosaics exhibiting a largely acicular shape.

3.2.12 *Mrozowski cracks, n*—a subset of accommodation cracks formed between basal planes within coke particle crystallites and the filler-binder phase from mismatches in thermal contraction of the graphite following cooling after graphitization (manufacture). These may also occur between crystallites if crystallite binding energies allow.

3.2.13 *optical domain, n*—the smallest region of local preferred orientation with relatively small misorientation angles appearing isochromatic under polarized light with a sensitive tint plate.

3.2.14 *optical texture, n*—fine structure in an optic array giving rise to color variations under polarized light, attributed to variations in the optic axis of domains.

3.2.15 *pore, n*—see *void*.

3.2.16 *porosity, n*—fraction of the total volume of a material occupied by both open and closed pores and cracks.

3.2.17 *void, n*—unfilled space enclosed within an apparently solid carbon or graphite body.

4. Significance and Use

4.1 The purpose of this guide is to provide a framework for consistent description of microstructural and microtextural features visible in optical micrographs of graphite. It also provides some guidance on sample preparation and image processing.

5. Optical Microscopy Methods

5.1 Three different methods of illumination are generally employed in optical microscopy: optical or bright field (BF), fluorescence under UV light, and polarized light. While bright field and polarized light methods can be undertaken directly on a prepared graphite surface, fluorescence requires the sample to be impregnated with a resin incorporating a fluorescent dye prior to preparation of the graphite surface. It is common for all three methods of illumination to be used in the characterization of graphite microstructure and texture so that resin impregnation is a standard procedure in sample preparation. It should also be noted that resin impregnation stabilizes the graphite matrix and protects porosity from dust intrusion during polishing of the surface being prepared for examination.

5.2 If the sample requires impregnation, a low-viscosity resin is used to impregnate and encapsulate the sample. The resin can have a small amount of fluorescent dye added for observation under ultraviolet (UV) light. Once impregnated with resin and cured, the encapsulated sample is ready for preparation of an examination face.

5.3 The selected face of the sample is prepared for microscopic examination by grinding it using progressively finer silicon carbide (SiC) papers to 2500 grit (8.4 µm ± 0.5 µm). The face is then further polished with a diamond suspension to a 1 µm finish. The same procedure is employed for both untreated and impregnated graphite samples. At this stage, the prepared face of the sample is ready for optical examination.

5.4 With BF illumination, the sample is observed using white light at normal incidence. Within the constraints of the optical resolution, this method of illumination allows microstructural features in the sample to be seen.

5.5 With fluorescence microscopy, incident UV light causes the dye in the resin to fluoresce, thus showing the extent of resin penetration into the sample and an indication of areas of open porosity. This method requires full impregnation of the accessible porosity by the resin, which can be influenced by the viscosity of the resin and extent of evacuation. The method is less revealing in terms of characterizing microstructure in fine-grained material because of incomplete penetration of the porosity by the resin.

5.6 Illumination with polarized light merits a more detailed explanation. The random variations in a light beam are in directions normal to the direction of propagating light. If the light beam is passed through an optically active crystalline material (a plane polarizer), some directions of vibrations will

be suppressed and others rotated. The net result is that specific directions of vibrations are favored on passing through the polarizer. If the transmitted plane-polarized light is examined after passing through a second optically active material, and this second optically active material is at right angles to the polarizer, then the light will be cut off completely. When the two optically active materials are in this position they are said to be crossed. The second optically active material is termed the analyzer. Polarization will occur on reflection from most crystalline materials, even when they are isotropic. Examination with crossed polarizers allows the polarization caused by interaction with the specimen surface to be studied. The degree of polarization will depend on the angle between the incident light and specific crystal planes in the material. Also, qualitative analysis of the specimen's surface relative crystallography and degree of crystallinity can be made.

5.6.1 If a sensitive tint plate is placed between the polarizer and analyzer, orientations of isotropic materials can be distinguished. A 1 λ plate is most commonly used but ½ λ plates may also be employed. A sensitive tint plate consists of a slice of some birefringent (birefringence is the difference between the highest and lowest refractive indices for anisotropic crystals) material that is cut parallel to the optic axis of the crystal. If plane-polarized light is transmitted through the sensitive tint plate, then the emergent ordinary and extraordinary rays will have a path difference of exactly one wavelength for light of one particular wavelength. In this case, the wavelength of green light is used, such that the transmitted light is white light minus the green wavelength, which is magenta in color.

5.6.2 A feature that appears dark with crossed polarizers will appear magenta with a sensitive tint plate in its 45º position. Other features with differing orientations and differing polarization characteristics will suppress other wavelengths and appear as a characteristic color (white light minus the suppressed wavelength). In this way, different orientations will produce different colors in cross-polarization optical microscopy. The strength of the colors observed will indicate the degree of long-range order and crystallinity within any one feature.

5.7 The specification for optical microscopy equipment will be determined by the resolution required to observe microstructural features of interest. Typically, low magnification images are taken with a 5× objective lens. The total magnification of the image will depend upon the strength of the ocular lens and the image capture arrangement. For more detailed images, objective lenses may range up to 100×, although image quality can be challenging at this level of magnification. Determination of optical texture can be influenced by the magnification employed, and the user should be aware that magnification requirements may differ depending upon the nature of the graphite under investigation.

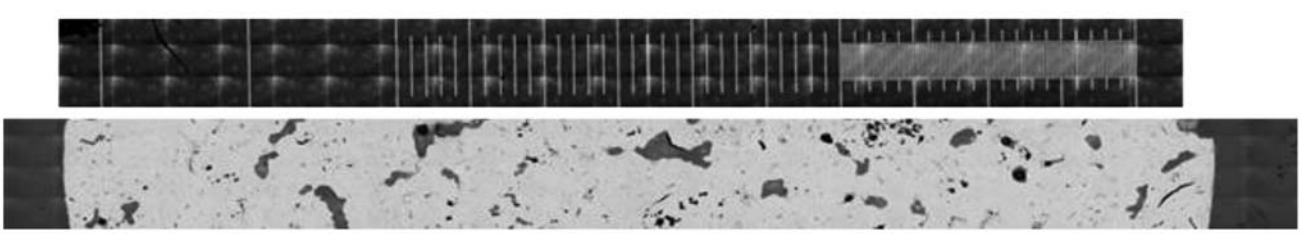
5.8 To correctly determine the size of optical objects or to measure distances on optical microscope images, or both, a spatial calibration must be performed. There are two basic ways to perform spatial calibrations: either by using known spatial references in the image, or through estimations based on camera and lens optical characteristics.

5.8.1 Calibrations based on known spatial references are more accurate, and should be used whenever trustful spatial references are available and can be imaged in identical optical conditions as the area of interest on the graphite specimen. When possible, a commercially available graduated reticle should be used as spatial reference. Using the tools available with modern microscopy image acquisition software, calibration should be done by repeatedly measuring linear segments drawn between known reference points on the reticle and saving the results along with the spatial distance values in appropriate units. If a graduated scale is not available, then any other object whose size can be accurately measured can serve as a spatial reference.

5.8.2 Fig. 1 shows an example of a graduated reticle. On top, a low magnification image shows a segment about 7 mm long of the graduated scale, with marks at every 1 mm (left), 0.1 mm (center) and 0.01 mm (right). The image is composed of 21 by 3 individual images stitched together. This image is useful for calibration of a series of equally large areas of interest on graphite specimens, acquired in exactly the same optical conditions as illustrated with the lower image in Fig. 1.

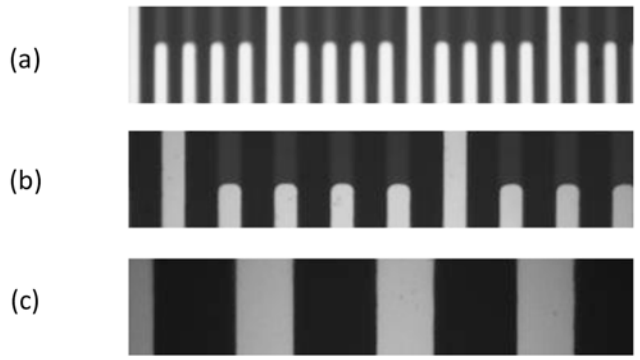
5.8.3 In Fig. 2, portions of the high density marks on the same reticle from Fig. 1 are shown, acquired with three different objectives (20×, 40×, and 100× magnification power). The shortest distance between the mark centers is 0.01 mm in all figures. These images can be used for spatial calibration of high magnification images acquired in identical conditions with each of the respective objective lenses.

5.8.4 Estimating the size of objects imaged by optical microscopy is also possible if the magnifying power of the objective lens and camera are known. For example, the image in Fig. 1 was collected using an objective lens with 40× magnification and a camera with 10× magnification power. The image recorded by the camera has a combined magnification equal to the product of magnification powers of the camera and objective lens (400× in the example of Fig. 1).



Upper image shows a 1 mm (left), 0.1 mm (center), and 0.01 mm scale; lower image shows an example optical micrograph.

FIG. 1 Use of a Graduated Reticle to Show Scale on Optical Micrographs



(a) 20×, (b) 40×, (c) 100×

FIG. 2 Magnification of a Graduated Reticle for High Magnification Images

5.9 The size of the specimen surface area covered by the image must be determined by the user. The low magnification image should be sufficiently large to be representative of the matrix, that is, contain repeatable microstructural features such as large filler particles and porosity. Such an image would then be used to select candidate microstructural features that might be examined at higher magnification. It is common for the low magnification image to be made up from a montage of smaller area images in order to be representative of the matrix. This is the case for the graphite image in Fig. 1. Alternatively, the entire surface of a specimen may be imaged in this way. Often an automated stepping stage will perform the compilation of the montage.

5.10 Examples of BF, fluorescence, and polarized light micrographs for a historical orthotropic needle coke nuclear graphite (pile grade A (PGA) graphite) are shown in Fig. 3. These montage images, taken using a 5× objective lens, provide a surface area of approximately 30 mm^2. As defined in Specification D7219, this graphite is categorized as a medium coarse-grained material because microscopic examination reveals no filler particles greater than 4 mm. In the BF image, dark areas signify porosity and the large filler particles can be clearly seen within the filler-binder phase. In the fluorescence image, the porosity in the BF image which has been impregnated with resin containing a fluorescent dye appears as light areas (the contrast of the BF image is reversed). A comparison of the two images will identify closed porosity, that is, porosity not accessible to the resin from the outside surface of the sample. The polarized light image, similar in contrast to the BF image, shows some variations in color due to different lamellar orientations.

5.11 Principal microstructural features that can be observed in BF and fluorescence micrographs are described in Section 7. Principal microtextural features that can be observed with polarized light micrographs are described in Section 8.

6. Origins of Texture in Graphite

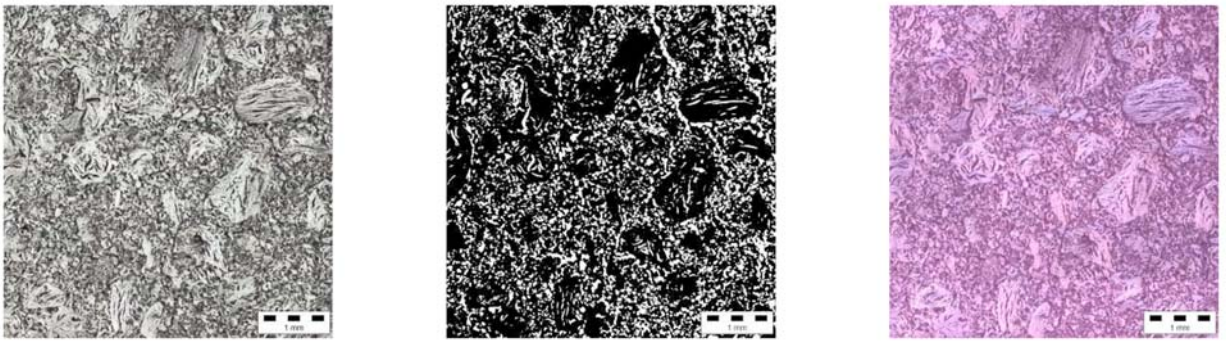
6.1 Synthetic graphite is formed from graphitizable carbons that have passed through a fluid (mesophase) during pyrolysis. Graphitization is a solid-state transformation of thermodynamically unstable pre-graphitic carbon into graphite by thermal activitation. The development of the mesophase (discotic nematic liquid crystal) during the fluid phase generates long-range ordering of polyaromatic molecules. The mesophase controls the basic microstructure of the final graphite material and hence its optical texture.

7. Microstructural Terminology for Graphite on the Optical Scale

7.1 A wide variety of terms are used for optically visible features in graphite. A simple scheme is presented in Fig. 4 showing what can be observed at different scales. The lowest level shaded in grey is at the nanoscale and not visible under optical microscopy.

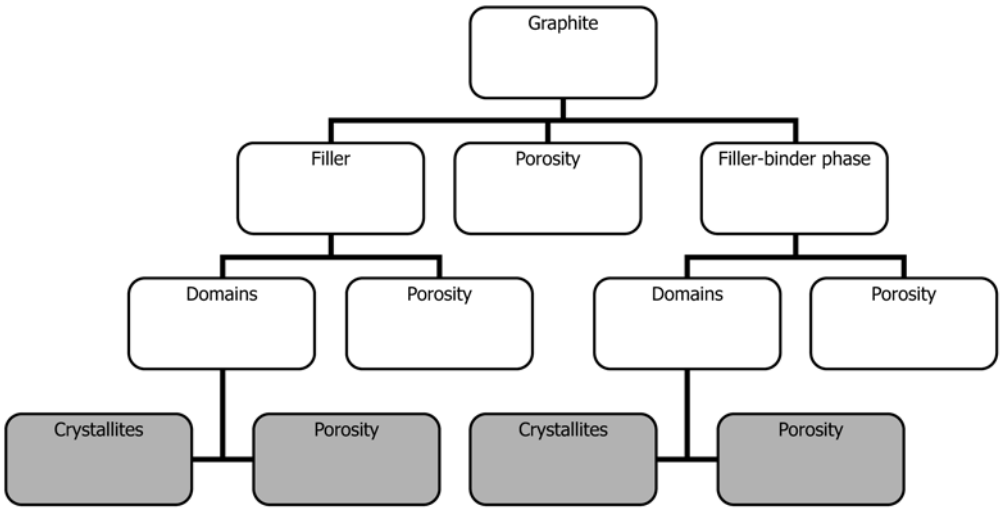
7.2 Although not universally adopted terminology, it is proposed that microstructure refers to the physical and spatial relation of the filler and binder matrix (porosity, pore size, etc.). Microtexture refers to the nature of the carbon in the matrix, its domain and crystallite development, degree of optical anisotropy, etc.

7.3 At the macroscale (first level down in Fig. 4), graphite can be separated into three phases: filler, porosity, and filler-binder phase. The filler is typically produced from coal tar pitch, crude oil derivatives, or naturally occurring asphaltenes in delayed coking refinery operations which are subsequently calcined. The filler can also be sourced as recycled or naturally occurring graphite. The filler-binder phase normally comprises very finely ground filler mixed with coal tar or petroleum pitch residues (binder). Porosity at this scale is the voidage within



BF (left image), fluorescence (center image, filler particle labeled A), and polarized light (right image).

FIG. 3 Micrographs of Medium Coarse-Grained Orthotropic Needle Coke Nuclear Graphite



Features in the greyed boxes are not visible on the optical scale.

FIG. 4 Schematic Showing Hierarchy of Principal Microstructural Terms for Graphite on the Optical Scale

the filler and filler-binder phases formed by incomplete local packing, shrinkage of the binder-coke mix on carbonization, and large-scale gaseous evolution channels arising from the release of gas and vapor decomposition products during manufacture.

7.4 At the microscale (second level down in Fig. 4), both the filler and filler-binder phase are each made up of separate domains and porosity.

7.4.1 Domains are the smallest regions of local preferred orientation with relatively small misorientation angles appearing as isochromatic units that can be viewed under polarized light in the optical microscope. Domains with large misorientation angles show up in different colors when viewed with a sensitive tint plate. Higher magnification of isochromatic regions can reveal finer chromatic structure associated with orientation of crystallite clusters, usually discussed in terms of microtexture or optical texture as described in Section 8.

7.4.2 The domains often contain the intercrystalline fissures arising from anisotropic contraction due to carbonization/graphitization densification and from anisotropic thermal contraction on cooling from thermal processing (Mrozowski cracks). Accommodation cracks are also produced throughout the graphite microstructure at domain interfaces in both the filler and filler-binder phase.

7.4.3 Much of the difference between graphites manifests itself at the domain level. The difference between the filler-binder phase and filler regions is largely in the domain texture: intra-domain porosity (fissures), orientation of porosity, and preferred orientation of layer planes within the domains (extent of wrinkling). There will be a connection between the structural parameters at the nanoscale and some of the domain parameters. For example, in nuclear graphites, coherence lengths are reduced by irradiation damage causing changes to local orientations within the domains.

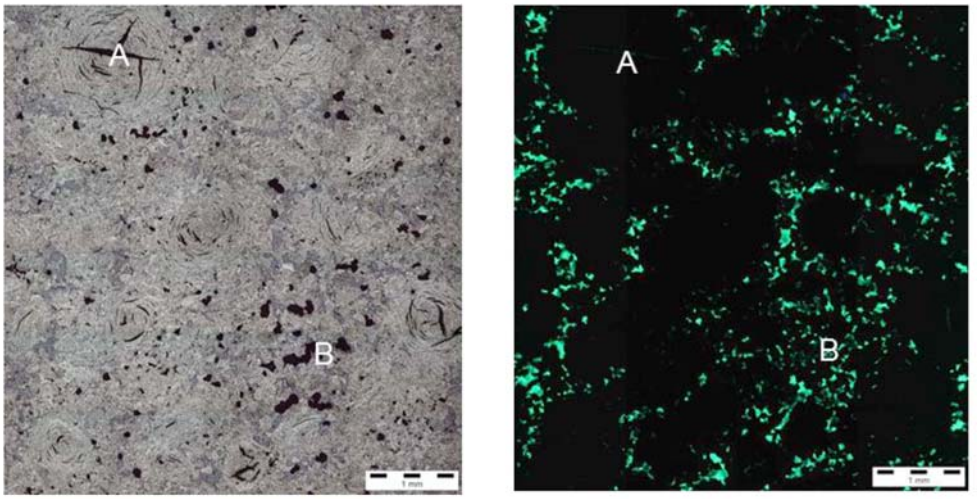
7.5 At the nanoscale (third level down in Fig. 4), domains in the filler and filler-binder phase can be further subdivided into crystallites and porosity, visible only by scanning electron microscopy and transmission electron microscopy. The crystallite can be regarded as a large region containing dislocations, which can be subdivided at an additional level corresponding to coherently diffracting regions (not shown in Fig. 4).

7.6 The principal microstructural features observed using BF and fluorescence micrographs, more commonly in coarse- and medium-grained graphites, are shown in Fig. 5 using image examples for a medium-grained, near-isotropic nuclear grade graphite (Gilsocarbon graphite). Unlike the orthotropic needle coke graphite (PGA graphite) shown in Fig. 3, the graphite illustrated in Fig. 5 is characterized by approximately spherical or oval filler coke particles. Large filler particles (example A in Fig. 5) show as dark areas in the fluorescence image indicating an absence of open porosity (at this resolution). From the BF image, large filler particles can be seen to contain closed porosity (dark areas). The areas surrounding the large filler particles comprise the filler-binder phase (example B in Fig. 5). By comparing the BF and fluorescence images, it can be seen that the filler-binder phase contains both open and closed porosity.

8. Microtextural Terminology for Graphite on the Optical Scale

8.1 Graphite formed from graphitizable carbon passes through a fluid phase (mesophase) during pyrolysis. Graphitization is a solid-state transformation of thermodynamically unstable nongraphitic carbon into graphite by thermal activation. The development of the mesophase during the fluid phase generates long-range ordering of molecules in linear arrays. The resulting microtexture gives rise to descriptive terms that include highly oriented particles (HOPs) and mosaics. Mosaics have historically[3] been subdivided according to their size and shape, giving rise to nomenclature such as very fine-grained, fine-grained, medium-grained and coarse-grained mosaics, mosaic clusters, mosaic ribbons, and supra mosaics. Mosaic

[3] Marsh, H., Calvert, C., and Bacha, J., "Structure and Formation of Shot Coke—A Microscopy Study," *Journal of Materials Science*, Vol 20, No. 1, 1985, pp. 289–302.



A: Filler particle; B: Filler-binder phase (Gilsocarbon graphite).

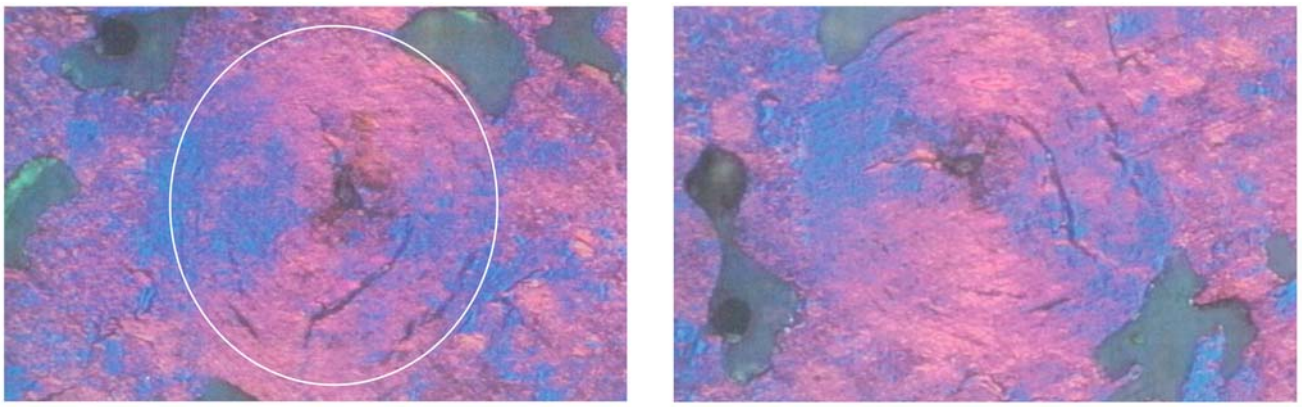
FIG. 5 Optical Micrographs of a Medium-Grained Nuclear Grade Graphite (Bright Field – Left-Hand Image, Fluorescence – Right-Hand Image)

features have been defined above, recognizing that they are not precise and are open to differing interpretations. Examples of some mosaic types are illustrated in the sub-sections below. The nomenclature for optical texture included in this guide is broadly consistent with that used for metallurgical cokes.

8.2 Microtextural features in the filler and filler-binder phases can be observed under polarized light. The principal features that may be observed are described in 8.3 – 8.6 with image examples for a medium-grained, near-isotropic nuclear grade graphite (Gilsocarbon graphite). Example micrographs for two other nuclear grade graphites have been included to provide a broader perspective on the types of image that may be encountered. These are the medium coarse-grained NBG-18 and the superfine-grained IG-110 graphite. The micrographs presented below have been compiled using different microscopy equipment at different laboratories and illustrate how the color and quality of images may vary but contain the same information.

8.3 Evidence for crystallinity in graphite can be found by optical imaging using polarized light. Fig. 6 shows two images of the same filler particle within the filler-binder phase, one image being rotated relative to the plane of the polarized light by 90°. The isochromatic color has altered position with respect to the specimen which demonstrates that the orientation of features changes with respect to the microscope stage, and hence the microstructure is crystalline in nature. Isochromatic areas of the images identify domains. These domains, indicating a preferred orientation of microstructural features at this magnification, have a fractal quality. Enlargement of isochromatic areas, either by magnification of the image (if resolution allows) or by reimaging using a higher magnification objective lens, can reveal further textural orientations with color patterns as referred to in 7.4.1.

8.4 Highly oriented particles (HOPs) are sometimes referred to in optical microscopy texts. These unidirectional granular agglomerates appear as uni- or bi-colored features under polarized light. They may either be filler particles with a strong preferred orientation or large elongated domains (and due to this uncertainty, no definition has been included above). HOPs can have a wide spread of shapes and sizes (typically mean



Specimen in right-hand image rotated 90 degrees relative to the plane of the light.

FIG. 6 Optical Micrograph of Gilsocarbon Filler Particle (Circled) Under Polarized Light

length 50 µm, mean width 10 µm). Examples of uni-colored and bi-colored HOPs are shown in Fig. 7.

8.5 Clusters of isochromatic units with group dimensions of the order of 10 µm to 500 µm are termed mosaics. Optically visible mosaics can take the form of isolated clusters (Fig. 8) or ribbons (Fig. 9), the latter reflecting the behavior of the fluid mesophase present during carbonization. These microtextural features are very common in filler particles, but are often present in the filler-binder phase at filler particle boundaries and occasionally as isolated features in the filler-binder phase (Fig. 10).

8.6 The term supra mosaic can be applied to an aligned region of coarse mosaics exhibiting a largely acicular shape (Fig. 11). These often occur at filler particle boundaries and also adjacent to mosaic clusters and ribbons.

8.7 Optical micrographs of medium coarse-grained NBG-18 and superfine-grained IG-110 (typical grain size 20 µm) graphites under polarized light are shown in Fig. 12 and Fig. 13, respectively. The NBG-18 graphite is shown at a single magnification with grain direction. The isotropic IG-110 graphite is shown at two magnifications.

8.7.1 Based upon a typical grain size of 1.6 mm for NBG-18 graphite, the image in Fig. 12 should contain approximately five to six filler particles. The division between filler particle and filler-binder phase is difficult to delineate as the field of view at this magnification does not provide a representative area of filler particles, filler-binder phase, and porosity. Furthermore, for this graphite, the microcrystallinity within the large filler particles makes it difficult to distinguish filler particles from the filler-binder phase. However, the micrograph shows a number of common features including a highly oriented filler particle segment, porosity, and mosaic texture.

8.7.2 In contrast, IG-110 graphite has an average filler particle size of 20 µm. The micrograph shown in Fig. 13(a) has the same magnification as that of the NBG-18 micrograph in Fig. 12. The image shows uniformly distributed fine porosity (dark regions) within a matrix of small filler particles and filler-binder phase. At this magnification, it is not possible to distinguish between filler particles and filler-binder phase. However, under higher magnification in Fig. 13(b), it is possible to identify large highly oriented needle coke filler particles, smaller filler particles, porosity, and mosaic texture.

8.7.3 These example micrographs show that an appropriate optical microscope setup needs to be selected to suit the type of graphite under investigation and the user's requirements.

9. Keywords

9.1 bright field; carbon; fluorescent light; graphite; microstructure; microtexture; optical microscopy; polarized light

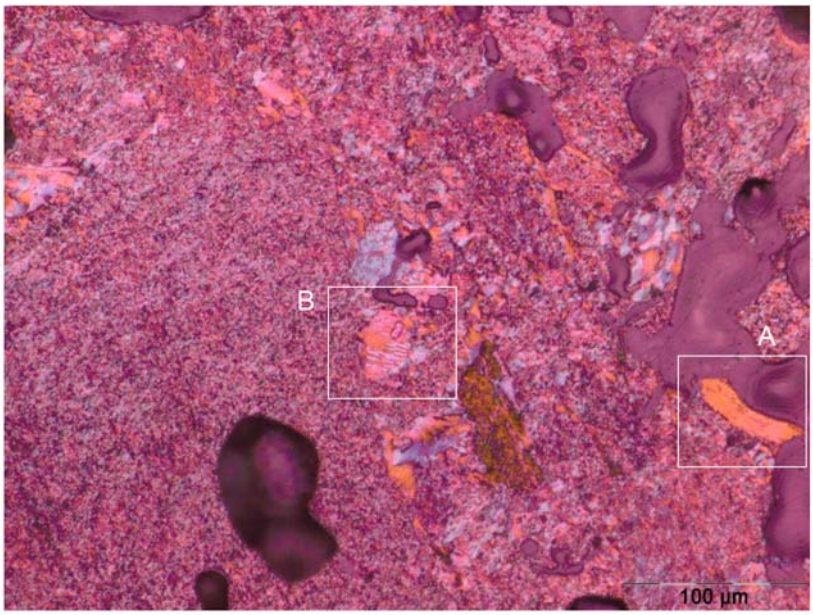


FIG. 7 Optical Micrograph Showing Uni-Colored Highly Oriented Particle (Box A) and Bi-Colored Highly Oriented Particle (Box B) Under Polarized Light (Gilsocarbon Graphite)