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

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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 and health practices and determine the applicability of regulatory limitations prior to use.*

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:

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.

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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.

3.2.7.4 *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 45° 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.

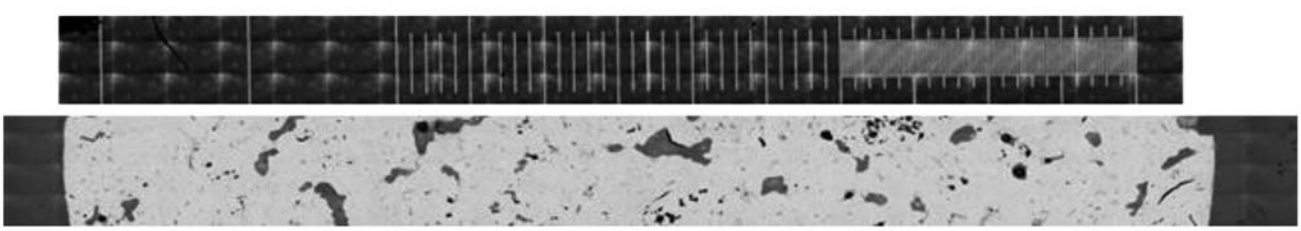
5.8.1 Calibrations based on known spatial references are more accurate, and should be used whenever trustful spatial references are available and can be imaged in identical optical conditions as the area of interest on the graphite specimen. When possible, a commercially available graduated reticle should 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).

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



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