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Standard Test Method for Determining Nodularity And Nodule Count In Ductile Iron Using Image Analysis[1]

This standard is issued under the fixed designation E2567; 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.

INTRODUCTION

Ductile cast iron, also known as nodular cast iron, and spherulitic or spheroidal graphitic iron, is produced with graphite in a spherulitic form. Nodularizing elements, such as magnesium, cerium, lithium, sodium etc., are added to a molten metal bath of proper chemical composition to produce discrete particles of spheroidal-shaped graphite. The control of graphite shape is critical to nodular iron properties. A reproducible measurement method is required for evaluation of the cast product and to control process variability. Shape is a difficult parameter to assess using standard chart methods, unless the shape is very close to well-recognized geometric shapes. Nodule density is also difficult to assess by chart methods as nodule size is also a variable and the chart cannot depict nodule density variations for nodules of all possible sizes. Stereological and metrological methods provide unbiased techniques for assessing structural variations. These procedures are best performed by image analysis systems that eliminate operator subjectivity, bias and inaccuracies associated with manual application of stereological and metrological methods. The metallographic sectioning plane will cut through the nodules at random, producing images of graphite nodules with circular or near-circular peripheries with a range of diameters.

1. Scope

1.1 This test method is used to determine the percent nodularity and the nodule count per unit area (that is, number of nodules per mm^2) using a light microscopical image of graphite in nodular cast iron. Images generated by other devices, such as a scanning electron microscope, are not specifically addressed, but can be utilized if the system is calibrated in both *x* and *y* directions.

1.2 Measurement of secondary or temper carbon in other types of cast iron, for example, malleable cast iron or in graphitic tool steels, is not specifically included in this standard because of the different graphite shapes and sizes inherent to such grades

1.3 This standard deals only with the recommended test method and nothing in it should be construed as defining or establishing limits of acceptability or fitness for purpose of the material tested.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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.*

1.6 *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]

A247 Test Method for Evaluating the Microstructure of Graphite in Iron Castings

E3 Guide for Preparation of Metallographic Specimens

E7 Terminology Relating to Metallography

[1] This test method is under the jurisdiction of ASTM Committee E04 on Metallography and is the direct responsibility of Subcommittee E04.14 on Quantitative Metallography.

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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. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, see Terminology E7.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *MFD*—Maximum Feret Diameter

3.2.2 *minimum size requirement*—the size threshold below which graphite particles are eliminated from the analysis.

3.2.3 *nodule*—a discrete graphite particle that exceeds both the required minimum size and shape factor as defined by this method.

3.2.4 *nodule count*—total number of graphite particles meeting the definition of a nodule in the area of interest (AOI).

3.2.5 *nodule density (Nodule count/unit area)*—number of nodules per mm^2.

3.2.6 *nodularity*—degree of roundness, or closeness to a circular periphery, of a graphite particle in ductile iron based upon the shape factor.

3.2.7 *percent nodularity by area*—the total area of particles defined as nodules which meet the minimum size requirements divided by the total area of all particles which meet the minimum size requirements, expressed as a percentage. See 8.10.

3.2.8 *shape factor*—a number between 0.00 and 1.0 resulting from formula (Eq 2) of this method.

3.2.9 *spherulitic graphite*—in cast iron, a small, spheroidal-shaped crystalline carbon body with a radial growth structure.

4. Summary of Test Method

4.1 This test method uses an image analyzer to measure the degree of roundness of graphite particles, viewed on a metallographic sectioning plane, that are above a minimum size in order to determine percent nodularity and nodule density. A common objective used for the analysis is 10×, for an overall magnification of approximately 100×. Higher magnification objectives (20× or more, for an overall approximate magnification of 200×) can be used to characterize small nodules, and a 5× magnification objective (approximate overall magnification of 50×) can be used to characterize very large nodules. The magnification used shall be in accordance to purchaser-supplier agreement and, shall be reported in the final report (see9.18). Threshold settings are established by the operator, and can be influenced by factors such as polishing technique, illumination intensity and uniformity, and lamp voltage and stability.

5. Significance and Use

5.1 Qualitative measurement of "nodularity" and "nodule count" using visual estimations has been practiced for many years. These methods suffer from poor reproducibility and repeatability. The introduction of computer-aided image analysis enables metallographers to measure and count individual particles of interest in a microstructure with a high degree of precision. This greatly reduces measurement variations compared to visual estimation methods (see, for example, Test Method A247).

5.2 This method defines a procedure for measuring the number of nodules and the quality of nodularity of spherulitic graphite in a cast iron microstructure. The specimen's location in a casting or cast test specimen, and the orientation of the plane-of-polish, are governed by product standards. When a product standard is not defined, choose the test location randomly or at specific systematically chosen depths as needed. The plane-of-polish may be parallel or perpendicular to the solidification direction, or chosen at random, depending upon the needs of the study.

5.3 This test method may be used to determine variations within a given test specimen, within a given location in a casting, between different locations in a casting, or for the same location in different castings over time. Results from this test method may be used to qualify material for shipment in accordance with guidelines agreed upon between purchaser and manufacturer or can be used to monitor process quality or product variations.

5.4 Measurements are performed using a computer-controlled automatic image analysis system.

5.5 A minimum number of specimens and a minimum surface area to be evaluated may be defined by producer-purchaser agreement, provided at least 500 particles meeting the minimum size requirements are measured. The number of particles analyzed shall be indicated in the final analysis report (see 9.6).

6. Test Specimens and Statistical Sampling

6.1 *Test Specimens:*

6.1.1 The number and location of test specimens, and the orientation of the plane-of-polish, should be defined by product standards or by producer-purchaser agreements. When this is not possible, the metallographer should use common-sense engineering analysis to decide on the number of specimens based upon the size of the casting, or the number of castings in the lot. The plane-of-polish may be chosen at random, or parallel or perpendicular to the solidification direction, depending upon the information required. The number and locations of test specimens, and the orientation of plane-of-polish, can be defined by product standards or producer-purchaser agreements.

6.1.2 Each specimen should have a surface area large enough to provide a number of fields-of-view at the required magnification. In general, a 10 mm × 10 mm surface area, or its equivalent area, is an acceptable approximate specimen size.

6.1.3 It is recommended to avoid sampling in the near-surface region, as this region will exhibit large variations in graphite structure as compared to areas further below the casting surface.

6.2 *Specimen Preparation:*

6.2.1 Metallographic specimen preparation must be carefully controlled to produce an acceptable quality surface for image analysis. Guidelines for preparing metallographic specimens are given in Guide E3.

6.2.2 Mounting of specimens is not required, but may facilitate identification coding or grinding and polishing.

6.2.3 The polishing procedure must remove all deformation and damage induced by the cutting and grinding procedure. Scratches and smeared graphite or matrix must be removed by final polishing with an abrasive of about 1 µm or less in size. Scratches must be small enough so as not to be detected during the thresholding step in the image analysis procedure.

6.2.4 Excessive relief between constituents on the plane-of-polish and excessive pitting or pullout of the soft graphite must be avoided. Graphite retention is critical. Specimens must be carefully cleaned and dried after polishing. Castings can contain shrinkage cavities and seepage of liquids or abrasives from these voids must be prevented.

6.2.5 Graphite shall be examined in the unetched condition. Etching the specimen is unnecessary and can lead to difficulties in segmenting the graphite from other microstructural features. It may not be possible to separate graphite from voids.

6.2.6 The preparation must be of sufficient quality to reveal the exact periphery of each nodule in the unetched condition.

6.3 *Statistical Sampling:*

6.3.1 At any specific test location, a minimum of 500 graphite particles should be analyzed for nodularity. In most cases it will be required to measure multiple fields of view to achieve the 500 particles per analysis minimum.

7. Calibration

7.1 Image analysis software includes a calibration procedure to determine the pixel size in the *X* and *Y* directions for each microscope objective (and any fixed zoom factors). If any linear or area data will be reported the system should be calibrated. Use a certified stage micrometer or one that is traceable to a certified stage micrometer, to determine the linear magnification for each objective (or for each objective and each available fixed zoom factor). To determine the pixel length in the *X* direction, the stage micrometer must be oriented so that its image appears as close to horizontal (0°) on the monitor as possible. When it is necessary to calibrate the pixel size in the *Y* direction, orient the stage micrometer on the stage so that the scale appears as close as possible to vertical on the monitor. Light intensity and focus should be adjusted to provide the sharpest image achievable. Always consult the manufacturer's operating manual for the specific calibration routine.

8. Procedure

8.1 Correct illumination is critical in all image analysis work, and measurement of graphite is no exception.

8.1.1 The lamp should be checked for correct alignment and the illumination intensity should be adjusted to the level required by the camera. Avoid saturating the camera. If a light over-flow indicator is available it must be used to set a constant light intensity. This will increase the repeatability of results from one analysis to another.

8.1.2 If a shading correction is available, it shall be used as it reduces light variations across the image; this improves thresholding.

8.2 Selection of magnification and measurement field is also of great importance.

8.2.1 At low magnification, survey the mount for homogeneity, polishing defects and abnormalities.

8.2.2 The default objective magnification used for the analysis is 10 ×which will produce an overall magnification of approximately 100×. A higher magnification objective (20× or more) can be used when characterizing microstructures with very small graphite particles. A 20× objective lens will produce and overall magnification of approximately 200×. Inversely, a lower magnification objective, such as 5×, can be used when analyzing very large graphite particles. A 5× objective will produce an overall magnification of approximately 50×. In all cases, the magnification selected shall be agreed upon via a purchaser-supplier agreement and, the magnification of the objective used for the analysis shall be indicated in the final report (see 9.18).

8.2.3 The industry norm for the minimum size to be considered as a nodule is 10 microns MFD. This limit is lower for thin walls castings. Other size limit can be used according to producer-purchaser agreements. Remove all particles from the analysis that have a MFD of less than 10 µm. This has the effect of removing noise, stray pixels, and smaller particles.

8.3 Well-defined particles should be visible on the monitor. Digitize the image to be analyzed. This image may have routines performed on it either to make the dark nodules darker or the white matrix whiter, but the shape and size of the nodule must be maintained. Smoothing or averaging filters that change the size or shape of a particle shall not be used.

8.4 The densitometer settings, or gray level threshold settings (or bit plane overlay), are chosen to segment the graphite particles of interest from the matrix. These threshold settings may result in detection of undesired features, such as voids, nitrides or inclusions. Adjust the threshold settings to minimize or prevent detection of these unwanted features. Some of the undesired constituents that are detected may be eliminated if they are smaller than the minimum size.

8.5 It is important to measure only whole particles. A properly sized guard frame shall be used to correctly measure graphite particles that intersect the edges of the measuring frame.

8.6 To qualify a graphite particle as a nodule its shape must meet or exceed a certain minimum shape factor value as defined in 8.8

8.7 Additional fields-of-view are evaluated until at least 500 individual particles are measured.

8.8 To determine if a graphite particle above the minimum size qualifies as a nodule, its shape must be quantified. Roundness or circularity will be assessed by use of a shape factor. A shape factor that does not require a perimeter measurement has been chosen, as the perimeter measurement is strongly influenced by magnification (pixel size) and by method used to measure the perimeter. For each particle, the area of a reference circle is calculated using the following equation:

$$Area\ of\ Reference\ Circle = \pi\ (Max.\ Feret)^2/4 \quad (1)$$

The shape factor for each graphite particle is then calculated from: