ASTM INTERNATIONAL

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

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

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

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

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

3.2.10 *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. The magnification used for the analysis is based on a Maximum Feret Diameter (MFD) $\geq$ 50 pixels and $\leq$ 125 pixels for the average size graphite particle being measured. Particles having a MFD <10% of the average MFD are not measured. 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. (See 5.6 for exceptions covering the minimum numbers of particles.)

5.6 The only exception to the 500-particle minimum assessment requirement is for large castings with a smallest cross-sectional dimension; for example being 15 cm. Graphite particles in such castings are very large typically above Test Method A247 size 4 or 160 µm, and nodule counts are low. The error in measuring a large graphite particle is very low. Therefore, when the mean graphite particle MFD is above Test Method A247 size 4 or 160 µm the number of particles required for a measurement may be reduced to 100 particles. However, the particles measured must still meet the minimum size requirement.

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 × 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