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Standard Guide for Additive Manufacturing of Metal — Finished Part Properties — Methods for Relative Density Measurement[1]

This standard is issued under the fixed designation F3637; 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 In this standard, guidelines for measuring post-manufacturing relative density of metallic additive manufactured (AM) parts and density assessment test specimens are given.

1.2 In this guide, standard test methods commonly used to measure part relative density and details any procedural changes or recommendations for use with PBF-LB parts are referenced. Extensibility to other types of metallic AM processes may be considered on a case-by-case basis with user discretion.

1.3 This guide is intended to be applied during the selection process of methods to measure the relative density of AM parts to balance cost, accuracy, complexity, part destruction, and part size concerns.

1.4 Pore size, shape, and distribution and their implications relative to the AM process and material are beyond the scope of this guide; however, each method's ability to obtain these metrics is discussed in the context of the various density measurement methods.

1.5 *Units*—The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard.

1.6 *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.*

1.7 *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]

B311 Test Method for Density of Powder Metallurgy (PM) Materials Containing Less Than Two Percent Porosity

B923 Test Method for Metal Powder Skeletal Density by Helium or Nitrogen Pycnometry

B962 Test Methods for Density of Compacted or Sintered Powder Metallurgy (PM) Products Using Archimedes' Principle

E3 Guide for Preparation of Metallographic Specimens

E494 Practice for Measuring Ultrasonic Velocity in Materials by Comparative Pulse-Echo Method

E1245 Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis

E1935 Test Method for Calibrating and Measuring CT Density

E2782 Guide for Measurement Systems Analysis (MSA)

F2971 Practice for Reporting Data for Test Specimens Prepared by Additive Manufacturing

2.2 *ISO Standard:*[2]

ISO/ASTM 52900 Additive Manufacturing — General Principles — Fundamentals and Vocabulary

3. Terminology

3.1 *Definitions*—Terminology relating to additive manufacturing in ISO/ASTM 52900 shall apply.

3.2 *Acronyms:*

3.2.1 *2D*—Two-dimensional

3.2.2 *3D*—Three-dimensional

3.2.3 *AM*—Additive manufacturing

3.2.4 *CAD*—Computer-aided design

3.2.5 *HIP*—Hot isostatic pressing

3.2.6 *LOF*—Lack of fusion

3.2.7 *NDT*—Nondestructive testing

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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.

3.2.8 *PBF-LB*—Powder bed fusion-laser beam

3.2.9 *XCT*—X-ray computed tomography

4. Summary of Guide

4.1 The relative density of a PBF-LB part, in the context of this guide, is expressed as a percentage relative to 100 % dense material (for example, 99.5 % density refers to the presence of 0.5 % observed porosity) or relative to a standard theoretical material density value (see 4.2 for further explanation). With respect to AM, relative density can be an indicator of process capability and resultant material quality. Density of a part, expressed as a percentage, is referred to as relative density within this guide.

4.1.1 Density traditionally takes on another meaning, specifically how much material, by mass, is contained within a certain volume. Realize that there are many different material density definitions, all of which are the mass of the material divided by its volume. It is the definition of the volume, and what is included in the volume, which differentiates the different material densities.

4.2 Some relative density measurements in this document rely on comparing the measured material density of the part, with units g/cm^3, to the theoretical material density, the standard measured value used to reference the true material density value. The intrinsic property of material density, with units g/cm^3 will not be used. Instead, material density references the measured material density of the part for the remainder of this document. Special care must be taken when selecting the theoretical material density value used in the calculation of relative density. It is recommended to use a trusted source, such as verified database, where theoretical material density values of sufficient precision can be obtained.

4.3 Different methods can be used to measure the relative density of finished AM parts. Relative density measurements are crucial in evaluating fabrication quality, as low-relative density values are indicative of process-related defects. In this guide, the following relative density measurement methods will be discussed in detail.

4.3.1 The Archimedes method measures material density by comparing the dry mass of a part and the submerged mass of the part. The measured material density is then compared with the theoretical material density to determine relative density.

4.3.2 Gas pycnometry measures the material density by the volume of gas displaced by the solid part and divides the mass of the part, determined with a separate measuring device, by the volume. This measurement is then compared to the theoretical material density to determine relative density.

4.3.3 XCT captures data from X-ray measurements at different angles. These data are reconstructed to determine relative density by identifying the process-induced defects in grayscale images and quantifying them in terms of voxel size.

4.3.4 Ultrasonic testing measures material density based on the velocity of ultrasonic waves passed through a part and reflected to the transmitter. The velocity measurement is used to calculate material density, which is then compared to theoretical material density to determine relative density.

4.3.5 Metallography and serial sectioning are destructive methods that captures 2D images of specimen sections. Relative density is calculated via area fraction of pores.

5. Significance and Use

5.1 *General:*

5.1.1 This guide is intended to support PBF-LB process and parameter development, part acceptance criteria, and process control tests.

5.1.2 *Flaws and Defects*—Fabricating fully dense parts continues to be a challenge in AM as the process intrinsically introduces volumetric flaws into a part reducing the part relative density (that is, increasing porosity or the presence of small voids in a part making it less than fully dense) and mechanical performance.

5.1.2.1 When a flaw reaches a size, shape, location, or criticality that makes it becomes unacceptable for part acceptance, it will be referred to as a defect.

5.1.2.2 Flaw or defect formation is governed by the manufacturing process, build parameters, feedstock, and geometric factors. Therefore, accurate measurement of fabricated part relative density is an important initial step in determining part and process quality.

5.1.2.3 The quantity, size, and shape of the volumetric flaws influences mechanical performance of a part, particularly under cyclic loading. These data could indicate irregularly shaped (for example, LOF pores or microcracking) or spherical porosity (for example, keyhole or entrapped gas porosity) and determine acceptability by assigning criteria. While these metrics can be quantified, in this guide, the general capabilities of each method to capture this data will be highlighted, but detailed recommendations on these data types will not be made and rather the focus will be on relative density measurements.

5.1.3 *Uncertainty and Error*—Users should consider that each measurement technique considered in this guide has differing sensitivities to various sized features. The measurement methods will also have different potential systematic errors or measurement uncertainties due to sampling sizes, detection resolution, effect of surface condition, experimental set-up, or reliance on a theoretical material density. It is important that these effects are taken into consideration as well as the natural statistical variability in the measurements. Multiple measurements of nominally identical test specimens should be made to enable the quantification of statistical uncertainty. Systematic uncertainty contributions will not be reduced by greater numbers of repeated measurements. When measuring specimens with relative densities close to 100 % quantification of systematic uncertainty for the selected measurement technique(s) becomes more critical to separate measurement and systematic variation from variation driven by the AM process. Differing levels of rigor can be applied when determining the role of uncertainty and variation depending on whether the measurement is in support of process development (for example, identifying appropriate fabrication parameters) or part acceptance (for example, part qualification).

5.1.4 *Repeatability and Reproducibility*—As uncertainty and error can be introduced into the measurement process through operator variation. Performing gage repeatability and

reproducibility (Gage R&R), a process that determines a test method's repeatability and reproducibility, is recommended for methods that rely on significant manual specimen preparation or operation such as Archimedes, pycnometry, ultrasonic, and metallography. Refer to Guide E2782 for guidance on performing this process evaluation.

5.2 *Method Selection:*

5.2.1 When evaluating methods, it may be beneficial to understand how the various attributes compare from method to method. In Fig. 1, a summary matrix comparing these various methods and their qualities is given.

5.2.2 *Using Multiple Methods*—It can be desirable to use multiple methods to determine relative density. For example, using low-resolution XCT to measure larger part flaws and metallography to identify the quantity of smaller process flaws could prove to be a highly useful way of producing accurate flaw data. Another approach to strengthen measurement accuracy is by implementing multiple methods that operate on similar principles, such as pycnometry and Archimedes.

5.2.3 *Non-destructive Methods*—Archimedes, ultrasonic, pycnometry, and XCT are nondestructive methods, while metallographic methods require part destruction to get relative density measurements. All the nondestructive methods can be used to characterize part relative density; however, as part size increases, these methods can become cumbersome to use. Archimedes requires a much larger and dedicated setup for relative density calculation that can be expensive for the appropriate accuracy but remains the least cost-intensive option, XCT and ultrasonic results are highly geometry and size dependent, and many pycnometry devices cannot handle larger part volumes (many pycnometers are equipped to handle specimen volumes of 1 cm^3 to 3.5 cm^3, however there are some that can handle up to 10 cm^3). While several of these methods may not be suitable for characterizing larger part volumes, all can provide relative density. Low-cost and quick measurement methods, such as Archimedes, can be used as a means of process development or data for statistical process control during production.

5.2.4 *Pore Morphology Data*—Metallographic and XCT methods can provide relative density measurements and specific geometric details (that is, size, aspect ratio, and shape) of individual flaws in addition to the overall part relative density. However, metallographic and XCT measurements are highly dependent on the resolution of the data, whether that is the sections examined, quantity of images, or microscope resolution, or a combination thereof, for metallographic methods or voxel size used for XCT. Archimedes, ultrasonic, and pycnometry methods do not provide these types of data when measuring relative density.

	NDT	Cost	Repeatability	Accuracy	Complexity of the technique	Geometry independent	Material independent	Size dependency	Speed	Uncertainty	Surface condition independent
Archimedes	↑	↑	↓	=	↑	=	↑	=	↑	=	=
XCT analysis	↑	↓	↑	↑	↓	=	=	=	=	=	=
Gas pycnometry	↑	=	↑	=	↑	↑	↑	↓	↑	=	=
Metallography	↓	=	=	=	=	=	↑	=	↓	=	↑
Ultrasonic	↑	↓	↓	↓	↓	↓	↓	↓	↑	↓	↓

↑ Green arrow indicates a desirable attribute of the test method

= Blue equal sign indicates a neutral attribute of the test method or that is possesses positive and negative attributes.

↓ Red arrow indicates an undesirable attribute of the test method

FIG. 1 Comparison Matrix of the Test Methods Evaluated in This Guide

5.2.5 *Relative Density Measurements Relying on Theoretical Material Density*—Archimedes, ultrasonic, and gas pycnometry methods rely on theoretical material density values in the calculation of relative density. The theoretical material density value selected is a possible source of systematic error. Material density is composition dependent. Each material will have a compositional specification and an allowable variation of that composition. This combined with material vaporization during fabrication could lead to a different material density value than the reported value by a material vendor or online source. The user should use caution on the reliance of a reported value and ensure the theoretical density is representative of the material (that is, from the specific material lot, measured from final material, or from a reliable database such).

5.2.5.1 For methods relying on comparing the measured and theoretical material densities to calculate the relative density of the specimen, the following formula should be used:

$$Relative\ Density(RD) = \left(\frac{measured\ material\ density}{theoretical\ material\ density} \right) \times 100\% \quad (1)$$

5.3 *Method Specific Recommendations:*

5.3.1 *Archimedes Method*—The Archimedes method is highly cost effective, nondestructive, and relatively non-geometry dependent; however, a significant amount of variation can be introduced into the process from the operator, part size, surface finish of the part, fluid entrapment, evaporation of fluid, temperature, water purity, absorbed gases, surface pores or cracks, and bubbles. Uncertainties of approximately 0.1 % for relative density measurements can be achieved for fully dense materials using this method. The sources of variation combined with part size will increase this uncertainty. However, training and consistent practices can minimize the effects of variation between measurements. Additionally, there are two main ASTM International standards for Archimedes measurements, Test Methods B962 and B311. Test Method B311 is specifically designed for measuring material density of parts with less than 2 % porosity volume and is, therefore, recommended as the measurement method for PBF-LB parts. The major difference between the two methods is that Test Methods B962 require fluid impregnation to deal with surface-connected porosity and Test Method B311 does not. If a specimen increases in mass while submerged in water, use Test Methods B962, and if the specimen does not gain mass, then Test Method B311 is applicable. Agitating the PBF-LB specimens while submerged is recommended to reduce any air pockets that may exist on the part's surface. Additionally, a benefit of AM is the ability to achieve high complexity—a potential source of error using this method would be internal channels or the ability for the liquid to cover the entire volume. It is recommended to take multiple measurements when using this method and compute a standard deviation.

5.3.2 *Gas Pycnometry Method*—Gas pycnometry requires that specimens be free of contaminants that may outgas during the test, shall not react with the displacing gas, and shall have sufficient strength to avoid deformation in the pressurized gas environment. Additionally, this method should only be used to measure parts with high relative densities since this method uses a gas to determine volume. Specimens with surface porosity or interconnected pore structures (whether through process defect or by design) will measure the skeletal volume, resulting in an inaccurate relative density measurement. This method functions on similar principles to that of Archimedes; however, it does not possess as many potential sources of error related to using a liquid for volume displacement. Uncertainty in this method is a function of part size and equipment capacity. There are several equations to calculate uncertainty from the equipment manufacturer; however, it will be equipment and part specific. Note that pycnometry determines a volume that can be compared directly to theoretical part volume based upon CAD dimensions of the part being produced. Differences can point directly to the volume of closed porosity in the produced part. Test Method B923 is used for measurement of skeletal or material density. While this test method is primarily for determination of skeletal density of metal powders, it has also been found to be useful for determination of skeletal volume and density of parts produced by traditional powder metallurgy methods. Note that it is best to try to choose a pycnometer capacity and specimen container configuration that result in the specimen under test occupying as much of the specimen container volume as possible. It can occur that a produced part is too large for any commercial gas pycnometers, and if so, another listed method shall be selected. Multiple measurements should be taken when using this method and compute a standard deviation.

5.3.3 *Ultrasonic Method*—Ultrasonic relative density characterization is limited in application by part geometry and suffers errors induced by small part sizes and surface roughness inherent in AM parts. Larger specimen parts with simple geometries (for example, cubes) and polished surfaces to measure from should be used for data capture. Note that this method is typically used as an NDT method and not for relative density measurement. Changes in measured velocity can be indicative of cracking or flaws within parts. These data received through ultrasonic testing can be evaluated into several equations provided in Practice E494 to calculate material density. This can then be compared to theoretical material density to compute a relative density measurement. However, several material constants such as Poisson's ratio or Young's modulus are required. To determine accurate values, additional testing is required, which can be cumbersome. Otherwise, vendor or online sources may need to be used to estimate these values, which may not be representative of the true values or include uncertainty considerations. Because of these factors combined with measurement variability inherent to measuring as-built AM parts, this is not a preferred method for measuring relative density of PBF-LB parts.

5.3.4 *XCT Method*—XCT provides highly descriptive data, such as pore size, shape, and distribution. However, it does require costly equipment and is time intensive. High-resolution (voxel size of ~1 µm to 5 µm) analysis is obtainable using XCT; however, there is a limitation on specimen size, requiring smaller parts, longer scanning times, and often more cost. Low resolution XCT can evaluate larger parts but is unable to detect fine details or smaller flaws. Successful parameters and software processing steps should be recorded to ensure repeatability. Additionally, the software tools used to filter noise and the