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Standard Guide for Additive Manufacturing – Feedstock – Particle Shape Image Analysis by Optical Photography to Identify and Quantify the Agglomerates/Satellites in Metal Powder Feedstock[1]

This standard is issued under the fixed designation F3571; 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 explains how to characterize the quality of metal powder feedstock to additive manufacturing (AM) relative to the powder shape using automated static or dynamic image analysis by optical photography. This guide will describe the method(s) to measure powder shape parameters that can identify potentially detrimental powder characteristics and specifically describe how to identify and quantify the proportion of agglomerates/satellites and other irregularly shaped non-spherical powder particles in a powder batch.

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

B215 Practices for Sampling Metal Powders

B243 Terminology of Powder Metallurgy

2.2 *ISO Standards:*[3]

ISO 9276-6 Representation of results of particle size analysis — Part 6: Descriptive and quantitative representation of particle shape and morphology

ISO 13322-1 Particle size analysis — Image analysis methods — Part 1: Static image analysis methods

ISO 13322-2 Particle size analysis — Image analysis methods — Part 2: Dynamic image analysis methods

ISO 14488 Particulate materials — Sampling and sample splitting for the determination of particulate properties

2.3 *ISO/ASTM Standard:*

ISO/ASTM 52900 Additive Manufacturing — General principles — Fundamentals and vocabulary

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms pertaining to this standard not otherwise listed in 3.2, *Definitions of Terms Specific to this Standard*, reference should be made to ISO/ASTM 52900.

3.1.2 For definition of terms pertaining to this standard not otherwise listed in 3.2, *Definitions of Terms Specific to this Standard*, reference should also be made to Terminology B243.

3.2 *Definitions of Terms Specific to This Standard:* (Key Parameters)

3.2.1 *aspect ratio, n*—parameter name that has the formula:

$$\text{aspect ratio} = x_{F\min}/x_{F\max}$$

where:

$x_{F\min}$ = shortest distance between parallel tangents, and
$x_{F\max}$ = longest distance between parallel tangents.

3.2.1.1 *Discussion*—All parameter names in this guide are given in ISO 9276-6. The parameter name may be different depending on the manufacturer of the analyzer, but it could be used if it has the same formula or a formula that correlates directly with it. Aspect ratio is on a scale of 0 to 1. A 2 by 4 shape has an aspect ratio of 0.5. A circle has an aspect ratio of 1. The parameter names B/L (breadth/length), and W/L aspect

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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] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

ratio are also used by some manufacturers for aspect ratio and correlate directly with $x_{F\min}/x_{F\max}$.

3.2.2 *convex hull, n*—The outer boundary of a particle image, as a rubber band would fit around the image as shown in Fig. 1. with the convex hull going from point to point around the star.

3.2.3 *ellipse ratio, n*—this parameter is given as $x_{L\min}/x_{L\max}$, where $x_{L\min}$ and $x_{L\max}$ are the lengths of the axes of the Legendre ellipse, whose calculation is given in Appendix X2.

3.2.3.1 *Discussion*—The axes of the Legendre ellipse are always perpendicular to each other, but the axes of aspect ratio ($x_{F\min}/x_{F\max}$) are not. They approach being perpendicular the higher the aspect ratio is, and the maximum deviation from perpendicular is for the shape of a square, where the aspect ratio is 1/1.414 (0.707), because the diagonal of a square (or any rectangle) is $x_{F\max}$. The ellipse ratio of a square is 1.0.

3.2.4 *solidity, n*—parameter name that has the formula:

$$\text{Area of the Particle/Area within the convex hull}(A / A_C)$$

3.2.4.1 *Discussion*—Solidity is on a scale of 0 to 1. This "star" particle (Fig. 1) would have a solidity less than 1. A value of 1 represents a particle whose area completely fills the area of the convex hull. As particles fill less and less of the area of the convex hull, the solidity value decreases and approaches 0. Some manufacturers use the parameter name convexity to be the square root of solidity, which correlates directly with solidity. [ISO 9276-6 defines convexity as the perimeter of the convex hull divided by the perimeter of the particle (P_C/P)].

4. Summary of Guide

4.1 There are two types of automated particle image analysis: static and dynamic. In this guide, both types use optical photography as the analytical technique. The operation of a static image analyzer by optical photography is described in ISO 13322-1. The operation of a dynamic image analyzer by optical microscopy is described in ISO 13322-2. In both cases, digitally photographed 2-D images of each particle in the measured sample are stored in an image file. After analysis, searches can be conducted to isolate and quantify various types of particles based on one or more size or shape parameters. Size and shape frequency distributions can also be reported, and the stored image file can be viewed for a visual understanding of the particle morphology.

4.2 Static image analysis (SIA) by optical photography is generally implemented on a research grade microscope fitted with either an automated stage driven by stepper motors that follow a chosen scanning pattern, or a manual stage with a controlled image pattern, plus a digital camera for photographing particles on the stage. A sample for measurement is dispersed in a vacuum-actuated device that disperses the sample particles across the stage. SIAs are usually used to measure dry samples, but some manufacturers also provide a wet cell option.

NOTE 1—SIAs use microscope objectives that cover up to five or six individual size ranges. Some manufacturers provide a software feature that can combine the image files measured on more than one objective, which gives the ability to measure broader distributions than only one objective would be capable of performing. The narrower the particle distribution and the smaller the mean value, the more particles can fit on the moving stage providing a more representative sample.

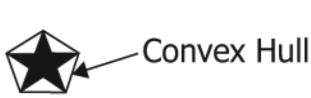

FIG. 1 Star Particle

4.3 Dynamic image analysis (DIA) by optical photography provides a wet or dry sample dispersion system that flows the particle stream between a high-speed pulsating source of light on one side and a digital camera on the other. Many size and shape measurements for each test can result in millions of particle images and records for analysis.

4.3.1 The standard wet DIA method generally recirculates the sample from a stirred dispersion module. Good method development practice would measure different amounts of samples for different time periods to reach a combination that gives high reproducibility on consecutive samples

4.4 The dry feeder DIA design generally provides a vibratory feeder to a drop point where the particles fall vertically through the sensing zone. There are sometimes free-fall (gravity fall) feeders plus an option for applying downward extra variable air pressure to disperse agglomerates that might be present. The dry feeder systems do not re-circulate material, and the sample can be fed to the vibratory feeder continually, and therefore, it can measure a much larger, more representative sample than a recirculating wet feed system can.

5. Significance and Use

5.1 Particle characterization, especially particle size distribution, has been an important parameter for quality control (QC) and research and development (R&D) in a very wide variety of industries and markets, anywhere a particulate system is a final product or an intermediate constituent somewhere in the process. But size alone is not a sufficient morphological measurement to use to understand many factors of the complete particle morphology of particulate systems and their effects on other properties. This information is expected to contribute to the understanding of the effects of shape on powder spreadability and flowability in the creation of the bed in powder bed fusion AM and the density and porosity of the final AM parts (definitions in ISO/ASTM 52900 and Terminology B243). Ultimately, specifications can be developed for quality control (QC) tolerances for these shape parameters that can be measured with a straightforward, fast automated analysis

6. Procedure

6.1 Extract a representative sample of metal powder feedstock based on recommended practices according to Practices B215 and ISO 14488.

6.2 Following the operator manual of the image analysis manufacturer, analyze the sample. Sample preparation, before and during analysis, and sampling procedure can be significant causes of error in particle characterization.

6.2.1 Sample preparation procedures for wet analyses of powder in DI water can include the addition of small amounts of detergents or surfactants to coat the particles preventing them from attaching to one other.

6.2.1.1 Also, most analyzers have ultrasonic transducers that can be used at various energy levels during wet analysis to help separate and disperse the particles.

6.2.2 For dry analyses, adjustable pressurized air can be used to provide the energy to separate the particles during measurement.

6.3 Following the search procedures of analyzer manufacturer's operator manual, identify and calculate the proportion of particles that have solidity and aspect ratio, or solidity and ellipse ratio parameters that are below either the AM manufacturer's specified tolerances or the tolerance guidelines in this guide.

NOTE 2—A Gage R&R, with an acceptable result on the applicable test method should be done before executing any testing described in this section to ensure consistent and accurate results.

6.4 As a guideline, measurements of a gas atomized metal powder have shown that election of a combination of aspect ratio and solidity, or ellipse ratio and solidity, can quantify the proportion of particles that are near perfect spheres, as well as those that are below those limits, the agglomerates/satellites or other irregularly shaped non-spherical particles. Results are given in Appendix X1.

7. Keywords

7.1 additive manufacturing; particle morphology; particle shape; particle size; powder; powder quality control

APPENDIXES

(Nonmandatory Information)

X1. DYNAMIC IMAGE ANALYZER (DIA)

X1.1 The following information follows from 6.4 which mentions analyses of a metal powder sample run on a DIA.

X1.1.1 In Fig. X1.1, three rows of the recorded image file are shown. The file is displaying the images in descending order by x_A per ISO 9276-6 (Da by most manufacturers), the area equivalent diameter. Ten of the images are fused agglomerates mixed in with good (round) individual particles, which shows the agglomerates would not be identifiable, separable, and quantifiable by size, but they can be by several shape parameters.

X1.1.2 By using the image analyzer manufacturer's "search" feature, one can enter limits on any parameter, above which identifies the round particles, and below which identifies the agglomerates/satellites and other non-spherical particles. These two classes can then be separated and quantified as proportions of the sample by both volume % and number %. Results by percentage of each type of particle are listed in Table X1.1. Two different pairs of shape parameters were used to isolate and quantify the round particles as well as the agglomerates/satellites and other non-spherical particles in a gas atomized metal powder: *(1)* aspect ratio with solidity and *(2)* ellipse ratio with solidity.

X1.1.3 The search function limit for aspect ratio was set for all particle images greater than 0.90, and for solidity, greater than 0.98. The search function limit for ellipse ratio was also set at greater than 0.90 and for solidity, greater than 0.98. The results from each search are the percentage of round (good) particles in the sample by volume and count (number).

X1.1.4 The percentage of round (good) particles along with the percentage, by subtraction, of non-round (bad) particles is shown in Table X1.1.

X1.1.5 The results in Table X1.1 show that there is no significant difference between using either aspect ratio or ellipse ratio in this case for atomized metal powders, which means there are no significant number of particles approaching a square shape. See Fig. X1.2. The results are not significantly different when limits on aspect ratio and solidity or ellipse ratio and solidity are used.

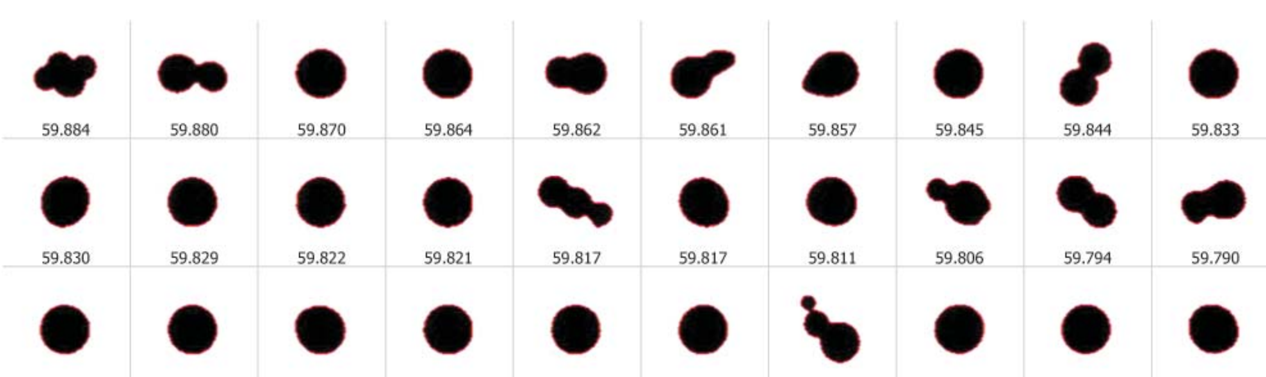

FIG. X1.1 Gas Atomized Metal Powder Particle Images in the Range of Da from 59.9 to 59.7