

INTERNATIONAL
STANDARD

ISO/ASTM
52907

First edition
2019-11

Additive manufacturing — Feedstock materials — Methods to characterize metal powders

Fabrication additive — Matières premières — Méthodes pour caractériser les poudres métalliques

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Reference number
ISO/ASTM 52907:2019(E)

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Published in Switzerland

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by ISO/TC 261, *Additive manufacturing*, in cooperation with ASTM F 42, *Additive manufacturing technologies*, on the basis of a partnership agreement between ISO and ASTM International with the aim to create a common set of ISO/ASTM standards on additive manufacturing.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The document aims to simplify the relation between the supplier and the customer for the supply of metallic powder for additive manufacturing purpose whatever the process involved.

The document does not aim to develop new standards but provides a list of existing standards dedicated to metallic powder that are suitable for additive manufacturing.

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Additive manufacturing — Feedstock materials — Methods to characterize metal powders

1 Scope

This document provides technical specifications for metallic powders intended to be used in additive manufacturing and covers the following aspects:

- documentation and traceability;
- sampling;
- particle size distribution;
- chemical composition;
- characteristic densities;
- morphology;
- flowability;
- contamination;
- packaging and storage.

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This document does not deal with safety aspects.

In addition, this document gives specific requirements for used metallic powders in additive manufacturing.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2591-1, *Test sieving — Part 1: Methods using test sieves of woven wire cloth and perforated metal plate*

ISO 3252, *Powder metallurgy — Vocabulary*

ISO 3923-1, *Metallic powders — Determination of apparent density — Part 1: Funnel method*

ISO 3923-2, *Metallic powders — Determination of apparent density — Part 2: Scott volumeter method*

ISO 3953, *Metallic powders — Determination of tap density*

ISO 3954, *Powders for powder metallurgical purposes — Sampling*

ISO 4497, *Metallic powders — Determination of particle size by dry sieving*

ISO 13320, *Particle size analysis — Laser diffraction methods*

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 22412, *Particle size analysis — Dynamic light scattering (DLS)*

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ISO/ASTM 52900, *Additive manufacturing — General principles — Fundamentals and vocabulary*

ASTM B212, *Standard Test Method for Apparent Density of Free-Flowing Metal powders Using the Hall Flowmeter Funnel*

ASTM B214, *Standard Test Method for Sieve Analysis of Metal powders*

ASTM B215, *Standard Practices for Sampling Metal powders*

ASTM B243, *Standard Terminology of Powder Metallurgy*

ASTM B329, *Standard Test Method for Apparent Density of Metal powders and Compounds Using the Scott Volumeter*

ASTM B417, *Standard Test Method for Apparent Density of Non-Free-Flowing Metal powders Using the Carney Funnel*

ASTM B527, *Standard Test Method for Tap Density of Metal powders and Compounds*

ASTM B822, *Standard Test Method for Particle Size Distribution of Metal powders and Related Compounds by Light Scattering*

EN 10204:2005, *Metallic products — Types of inspection documents*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 3252, ISO/ASTM 52900, ASTM B243 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1

EDX

X-ray spectrometry in which the energy of individual photons is measured by a parallel detector and used to build up a histogram representing the distribution of X-rays with energy

[SOURCE: ISO/TS 80004-13:2017, 3.3.2.4, modified — "EDX" has been kept as the only term and "are" has been changed to "is"]

4 Technical specifications

4.1 General

The supplier and customer shall choose the test methods appropriate to the customer's requirements.

4.2 Documentation and traceability

To ensure traceability, statements of conformity and inspection documents shall specify the following:

- a unique document reference,
- the name and the address of the supplier,
- the reference of powder lot,
- the product description, including chemical composition, standard and/or trade/common name,

- the nature of powder production process (including e.g. type of gas used, environment conditions),
- the packaging description, including the packaging, the nature of the shielding gas and the desiccant bag, if relevant,
- the date of analysis,
- storage and preservation instructions,
- all of the information to ensure the traceability (e.g. order number, applicable specification).

NOTE 1 When a desiccant bag is in contact with the powder, it can be a source of contamination.

The inspection document shall comply with EN 10204:2005, 4.1.

The statement of conformity should follow ISO/IEC 17050-1.

The reported values shall be linked to the test method used and the corresponding standard. The relevant standards to characterize metallic powder or feedstock for additive manufacturing are detailed in this document. Powder characteristics shall be subjected to a prior customer/supplier agreement.

The product shall be supplied with its material safety data sheet (SDS).

NOTE 2 The inspection document and the statement of conformity can be on the same document.

EXAMPLES Product description: Ni alloy 718 powder 10 µm to 45 µm.

Nature of production process: Vacuum Induction Melting argon gas atomization.

Packaging description: 10 kg bottle under Argon protective atmosphere.

For an example of certificate, see [Annex B](#).

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4.3 Sampling

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Samples shall be representative of the powder lot, ensuring homogeneity when split. Methods and equipment shall follow the requirements in ISO 3954, ASTM B215 or another method subjected to a prior customer/supplier agreement with the method(s) reported.

Procedures should be included for equipment cleanliness prior to sampling to prevent cross contamination of powder.

4.4 Particle size distribution

Particle size and particle size distribution shall be determined in accordance with one or several of the methods and standards listed in [Table 1](#).

The standards according to the methods used shall be indicated in the report (see [Annex B](#)).

Table 1 — Methods used for particle size analysis

Method	Typical range (varies between instruments)	Expression of results	Advantages	Limitations
Laser diffraction (ISO 13320)	0,1 µm to 3 mm	Cumulative volume percentages on a plot, with calculated values D_x at which X % of the total volume is below this value	<ul style="list-style-type: none"> — Ease of use — Large sample sizes are not required — High accuracy and repeatability 	<ul style="list-style-type: none"> — Measurements shall be made on isolated particles (not touching) – liquid suspension can be necessary — Assumes spherical particles when calculating volumes
Light scattering (ISO 22412, ASTM B822)	1 nm to 0,1 mm			
Image analysis (static: ISO 13322-1)	≥5 µm	Either the number or volume of particles in each size interval	<ul style="list-style-type: none"> — Accounts for non-spherical particles — Capable of reporting shape factors 	<ul style="list-style-type: none"> — Measurements shall be made on isolated particles (not touching) – liquid suspension can be used — Result accuracy depends on number of pixels — Set up procedures to be determined by the operator to achieve optimum results for that specific powder
Image analysis (dynamic: ISO 13322-2)	≥5 µm	Either the number or volume of particles in each size interval and shape distribution	<ul style="list-style-type: none"> — Accounts for non-spherical particles — Capable of reporting shape factors — Large amount of particles can be measured in each sample, which provides a large dataset for statistical analysis 	<ul style="list-style-type: none"> — Measurements shall be made on isolated particles (not touching) – liquid suspension can be necessary — Result accuracy depends on number of pixels — Set up procedures to be determined by the operator to achieve optimum results for that specific powder

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Table 1 (continued)

Method	Typical range (varies between instruments)	Expression of results	Advantages	Limitations
Sieving (ISO 2591-1, ISO 4497, ASTM B214)	≥45 µm	The amounts of particles present in specified particle size intervals, expressed as a percentage of the total particles	— Equipment required is cheaper overall than equipment required for other particle size distribution testing methods	<ul style="list-style-type: none"> — Appropriate sieve sizes required — Results obtained assume near spherical particles that pass through the sieve openings when the particle diameter is smaller than the size openings. This does not take into account long particles or particles with agglomerations. — Carefully controlled cleaning of sieves between tests required — Results obtained are discrete intervals — Not suitable for particles sized wholly or mostly under 45 µm

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NOTE 1 The laser diffraction and dynamic image analysis method results in higher values than the sieving method. The sieving method gives a weight percent of powder impeded by or passing through a square net whereas the laser diffraction method gives an equivalent diameter calculated from laser interferences.

NOTE 2 The results can be obtained by different methods; for example, quantification of the largest particles by sieving and quantification of the thinnest particles by laser diffraction. The results obtained from different methods can produce different results.

NOTE 3 The results are often expressed in D10 (first decile, i.e. 1/10 of the statistical population is below this value), D50 (median value, i.e. half of the statistical population is below this value) and D90 (last decile, i.e. 9/10 of the statistical population is below this value).

NOTE 4 Generally, the particle size distribution of used powder shifts with use. The degree of shift is a function of multiple parameters including material and process.

NOTE 5 Particle size is considered to be a useful property for comparing and controlling different lots of powder over time as this characteristic will change with powder use.

4.5 Chemical composition

The chemical composition of the metallic powder shall be determined by any suitable testing procedure, e.g. wet chemical processes, atomic absorption spectrometry, flame emission spectroscopy, or X-ray fluorescence analysis.

For powder mixtures, preparation of the sample for chemical analysis shall be performed in accordance with recognised methods.

NOTE 1 Example of recognised methods are available in MPIF STM 67.