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Additive manufacturing of metal — Feedstock materials — Powder life cycle management

Fabrication additive de métaux — Matières premières — Gestion du cycle de vie de la poudre

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Foreword

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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).

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This document was prepared by Technical Committee ISO/TC 261, *Additive manufacturing*, in cooperation with ASTM Committee F42, *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, and in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 438, *Additive manufacturing,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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

Metal powders represent the feedstock for numerous additive manufacturing processes. Specifications and quality of metal powder feedstock are directly related to the quality and performance of components fabricated by additive manufacturing (AM).

During their usage in additive manufacturing processes as well as during storage and handling, powders can be subject to various quality-relevant influencing factors.

These may include:

— cross-contamination and impurities;

— changes in particle size distribution;

— reactions with ambient gases;

— changes in moisture content;

— changes in flow properties;

— changes of particle morphology;

— absorption of welding fumes and spatters;

— changes in chemical composition due to selective evaporation of individual alloying elements.

Quality assurance of the powder materials over the entire service life from receiving, over storage and handling to reuse and disposal is therefore decisive for qualified additive manufacturing processes in any relevant industry.

This document aims to raise awareness of powder quality issues and to describe measures and procedures for quality assurance, batch identification and traceability of powder materials. The proposed measures are derived from best practices in the processing industry with a main emphasis on how frequently and at which stages of the process chain to document certain properties.

NOTE As the metal powder/feedstock is the main input of the AM process, its quality, both incoming and in service, impacts the quality of the AM output. However, the control over the quality of the input is one possible strategy to ensure the quality of the process output. Alternatively, the supplier/manufacturer is allowed to certify the quality of the AM components through

a) validation and verification of the AM process, as per internal procedures, and

b) inspection of the CTQs (critical to quality) of the AM components, as per customer agreement.

Additive manufacturing of metal — Feedstock materials — Powder life cycle management

1 Scope

This document specifies requirements and describes aspects for the lifecycle management of metal feedstock materials for powder based additive manufacturing processes. These aspects include but are not limited to:

— powder properties;

— powder lifecycle;

— test methods;

— powder quality assurance.

This document supplements ISO/ASTM 52907, which primarily focuses on requirements for virgin powder. This document covers on powder life cycle management, and therefore focuses on control of virgin and used powders.

This document can be used by manufacturers of metal powders, purchasers of powder feedstock for additive manufacturing, those responsible for the quality assurance of additively manufactured parts and suppliers of measurement and testing equipment for characterizing metal powders for use in powder-based additive manufacturing processes.

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 12154, *Determination of density by volumetric displacement — Skeleton density by gas pycnometry*

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

ISO/ASTM 52907, *Additive manufacturing — Feedstock materials — Methods to characterize metal powders*

ASTM B923, *Standard Test Method for Metal Powder Skeletal Density by Helium or Nitrogen Pycnometry*

3 Terms and definitions

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

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
split
action of physically or systematically splitting a batch into one or more smaller volumes of powder

Note 1 to entry: It is possible that such action is taken in order to differentiate between volumes of powder used for individual machines but which originate from a single large batch.

3.2
sub-batch
quantity of powder which has been *split* (3.1) from a larger batch

Note 1 to entry: A sub-batch can also be designated as a new single batch (e.g. with a sub-name or suffix).

3.3
combine
merge two or more powder batches of the same nominal specification in the same container or AM system without active blending

EXAMPLE An AM machine feedstock hopper is topped up with powder whilst an existing volume of powder remains in the hopper.

3.4
reuse metric
quantitative measure of the exposure or use of a powder batch in an AM process

Note 1 to entry: This may be expressed iteratively, for example a number of builds or exposures, or with a continuous scale such as total laser exposure time (laser-on time) or total incident energy.

4 Symbols and abbreviations

The following symbols and abbreviations are used throughout this document.

Table 1 — Symbols

Symbol	Designation	Unit
D_{V10}	10 % quantile of particle size based on the sample volume	µm
D_{V50}	50 % quantile of particle size based on the sample volume	µm
D_{V90}	90 % quantile of particle size based on the sample volume	µm
H	Hausner ratio	—
I_e	Inter-particle porosity	—
V_{drum}	bulk volume inside the drum	ml
V_{tap}	tapped volume	ml
ρ_b	bulk density	g/ml
ρ_{tap}	tapped density	g/ml

Table 2 — Abbreviations

Abbreviation	Designation
AAS	Atom absorption spectrometry
CoA	Certificate of analysis
DRIFTS	Diffuse reflectance infrared Fourier transform spectroscopy
EDX	Energy-dispersive X-ray spectroscopy
ELI	Extra low interstitial
ETAAS	Electrothermal atom absorption spectrometry
FAAS	Flame atom absorption spectrometry
GFAAS	Graphite furnace atom absorption spectrometry
ICP-OES	Inductively coupled plasma optical emission spectrometry
IoT	Internet of things
ppm	Parts per million
SEM	Scanning electron microscopy
XFA	X-ray fluorescence analysis
XRF	X-ray fluorescence spectroscopy

5 Powder properties

5.1 General

Powder properties are critical to the manufacturing process and the quality of the formed material in the final product. To help with the repeatability of the process and the quality and consistency of the products produced, certain powder properties require measurement and monitoring on receipt and re-use of the feedstock.

The following sub-clauses provide further information on these properties and the effects they have on the AM process and final product. Measurement techniques for each property are provided in each subclause.

For the process-influencing parameters discussed in the following sub-clauses, properties shall be determined by examining a representative sample of the powder, ensuring homogeneity when split. Refer to 7.3 for detailed information on sampling methods.

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

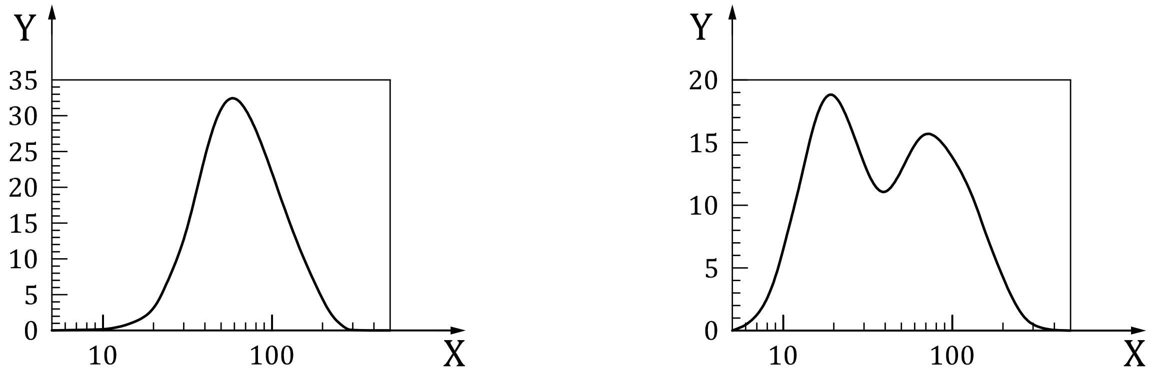
5.2 Particle size distribution

5.2.1 General

The particle size distribution of a powder is a set of characteristic values or a mathematical function that describes the relative amount of particles (typically by mass or number) of a certain size class or category.

During processing in additive manufacturing machines, the particle size distribution typically shifts to higher (coarser) values as small particles tend to be carried away by the inert gas flow and collected in ultra-fine filters, particles melt together to form larger particles, and reactions with ambient gases lead to surface layers on the particles, which increases their volume and mass.

The processing behaviour (e.g. flowability, packing behaviour) of a certain powder is particularly influenced by the width, the median and the lower and upper limits of the particle size distribution. Engineered powder lots can contain unimodal, bi-modal or multi-modal distributions (see Figure 1) with significant changes based on deviations from as-designed particle size distributions.



a) **Uni-modal** b) **Bi-modal**

Key

X particle size, in μm

Y relative proportion of particles, in %

Figure 1 — Particle size distribution charts

Particle size distribution has a direct effect on the processability of the powder. A shift in distribution which increases the proportion of fine or coarse particles has an impact on the flow characteristics and coating capacity of powder, for example in a beam melting machine. Particle size distribution also affects the density of the powder bed and thus its energy absorption and distribution behaviour.

The following methods may be used to determine the particle size distribution of particles in the metal powder. The latter methods mostly require greater analytical effort.

NOTE The results obtained from the different methods are in general not directly comparable.

5.2.2 Dynamic image analysis

Dynamic image analysis, in accordance with ISO 13322-2, is a method of characterizing particles by optically analysing their shadow projections. By capturing an image of the particle, it is possible to calculate various particle size and shape parameters such as diameter, maximum length, circularity, or minimum width. During analysis, each separate measurement is assigned to a measuring class according to its size. The upper and lower limit of the measuring range are determined by the camera resolution and magnification.

NOTE 1 The results of dynamic image analysis largely depend on the measurement parameters and settings of the evaluation algorithms. Consequently, significantly different results can be obtained from measurement systems made by different manufacturers.

It shall be observed that the results may be presented by volume or number.

NOTE 2 With these types of analytical systems, it is often possible to analyse particle size distribution and particle morphology simultaneously, allowing correlations to be made between morphology and particle size.

5.2.3 Laser diffraction and light scattering

Laser diffraction and light scattering in accordance with ISO 13320 or ASTM B822 calculates the particle size by measuring the light scatter produced (angle, intensity) as a laser beam passes through a dispersed powder sample. With laser diffraction, it shall be observed that the particle size distribution can be reported as volume- or number-based data. This method is used for particle sizes ranging from 0,1 μm to 3 mm and is highly suitable for comparative measurements. With irregularly shaped powder particles, for example, the particle size distribution obtained corresponds to the light scattering behaviour of a volume of spherical powder. Consequently, the results can differ from those obtained by dry sieving or sedimentation.

NOTE The results of laser diffraction largely depend on the measurement parameters and settings of the evaluation algorithms. Consequently, significantly different results can be obtained from measurement systems made by different manufacturers.

5.2.4 Dry sieving

Dry sieving in accordance with ISO 2591-1, ISO 4497 or ASTM B214 is only suitable for dry powders that contain no binders or auxiliary materials. ISO 4497 does not recommend the use of dry sieving for irregular powder particles or powders in which all or most of the particles have a grain size less than 45 µm.

5.2.5 Light or scanning electron microscopy (SEM) images

This method is already used in industry and research and can be applied synergistically to particle morphology analysis (see 5.6).

NOTE The number of particles captured and subsequently analysed by image processing software is limited and it is possible that it is not representative of the entire powder sample.

5.3 Chemical composition

5.3.1 General

The chemical composition relates to the relative amount of elements that constitute a powder or bulk material. Values are given either in atomic percent (at%) or mass percent (*m*%). Material properties are directly related to the chemical composition.

In manufacturing practice, the chemical composition of metal powder feedstock is subject to variation due to process-related phenomena, such as selective evaporation of individual alloying elements, reactions with ambient gases (e.g. oxygen, nitrogen) and/or absorption of fumes and spatters (see example for powder bed fusion in Figure 2).