International
Standard

ISO/ASTM 52928

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

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

First edition
2024-05

Reference number
ISO/ASTM 52928:2024(en)

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Published in Switzerland

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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 subclauses 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 subclauses, 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.

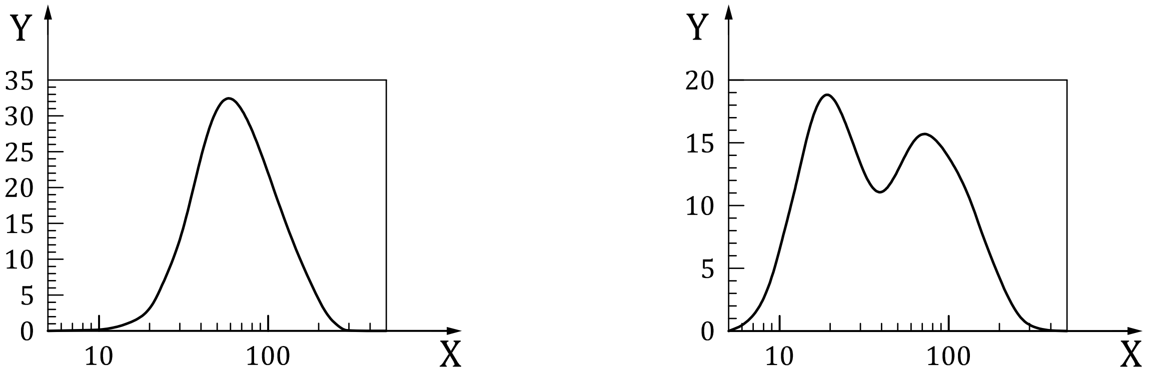
5.2 Particle size distribution

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 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).

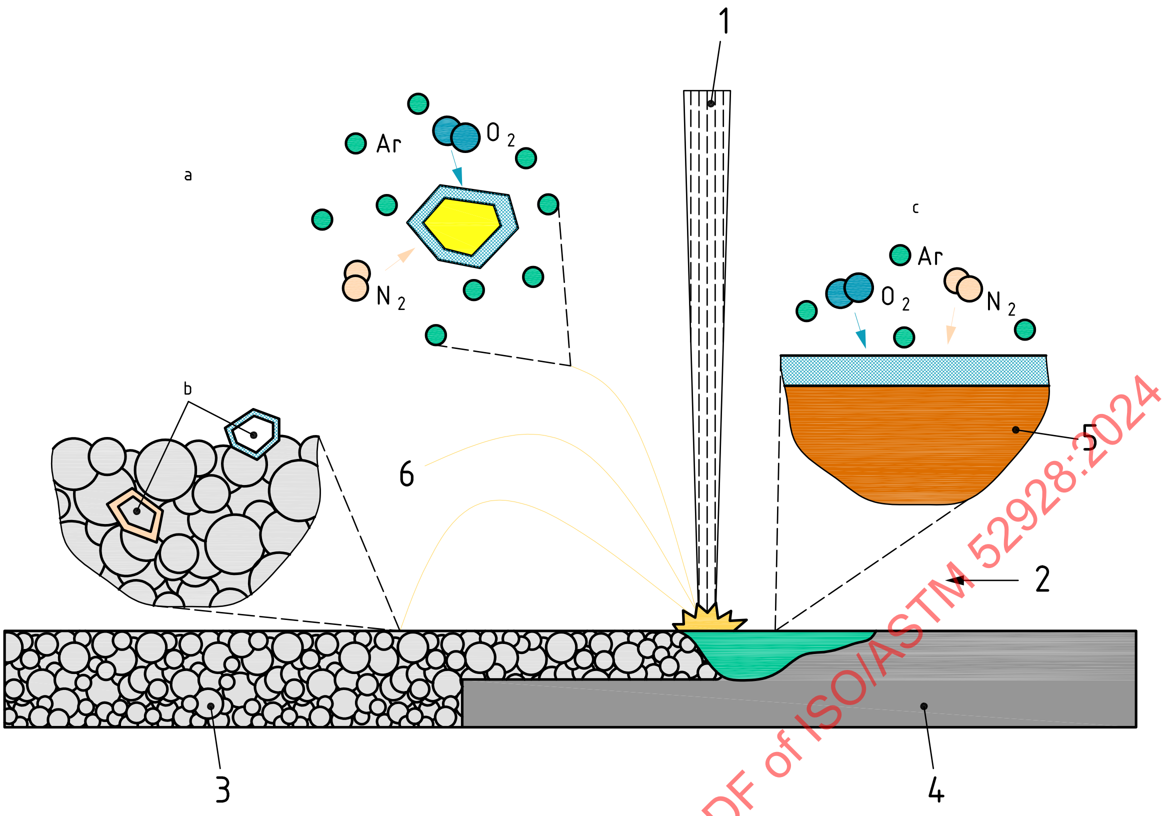
NOTE The number of particles captured and subsequently analysed by image processing software is limited and it is possible that it is not representative of the entire powder sample.

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).



Key

1	laser beam
2	shielding gas flow (direction is indicative only and can be in other directions, relative to beam travel)
3	powder bed
4	solid
5	melt pool
6	spatter
a	Spatter reacts with O_2 and N_2, forming O- and N-rich particles which deposit into the powder bed.
b	O- and N-rich particles.
c	O_2 and N_2 in protective gas atmosphere react with melt pool surface.

Figure 2 — Impact of spatter during build and effect on chemical composition

The additive manufacturer shall ensure that the chemical composition meets the powder specification limits.

NOTE 1 The powder specification can include chemical composition requirements that differ from those in the consolidated material specification to compensate for loss or gain of elements during the additive manufacturing process (e.g. over alloying).

The methods of 5.3.2 to 5.3.6 may be used to determine the chemical composition of the powder samples.

NOTE 2 Refer to ISO/ASTM 52907:2019, Table 2 for further details of suitable test methods.

5.3.2 Combustion methods

Combustion analysis is currently the best available method for analysing the chemical elements C, H, N, and S. After initial weighing, the sample is catalytically combusted with pure oxygen at high temperatures (up to 1 800 °C in an exothermic reaction). The combustion gases produced are then transported in a carrier gas (usually pure helium) through a copper or tungsten contact which is heated to approximately 600 °C to 900 °C. Nitrogen oxides (NO*x*) contained in the gas stream are completely reduced to molecular nitrogen

(N_2). Then the defined combustion gases (CO_2, H_2O, SO_2, N_2) are separated in specific separation columns or by gas chromatography and successively fed to a thermal conductivity detector where they are quantified.

With this measurement method, the peaks in the measurement signal can be clearly assigned to the elements under investigation. Analysis of the peak areas permits a quantitative determination of the individual elements. Using the known initial mass, the respective mass fraction (in percent or ppm) of the elements C, H, N, and S in the analysed sample can be precisely calculated.

The carrier-gas heat extraction process is also a combustion method.

5.3.3 Flame AAS

Atom absorption spectrometry (AAS) is an analytical technique that measures the absorption of radiation (light) by free atoms in the gaseous phase.

The oldest method is flame AAS (FAAS). The sample solution is aspirated into a burner, atomized and vaporized in the flame. Graphite furnace AAS (GFAAS), also known as electrothermal AAS (ETAAS), is a similar method in which the sample is atomized in a glowing graphite tube. Both processes are universally applicable.

5.3.4 X-ray fluorescence spectroscopy (XRF)

XRF, which is also referred to as X-ray fluorescence analysis (XFA) is one of the most widely used methods for the quantitative and qualitative analysis of the elements in a sample because it is non-destructive and does not require solutions or dilutions. The limit of detection is approximately one microgram per gram (i.e. 1 ppm[1]).

5.3.5 Inductively coupled plasma optical emission spectrometry (ICP-OES)

This technique is used for the quantitative and qualitative elemental analysis of solid, liquid, and gaseous samples. It is based on the principle that excited atoms emit electromagnetic radiation at wavelengths characteristic for a specific chemical element, thus providing information about the composition of the sample. The atoms are excited by an inductively coupled plasma (ICP) and the conversion into the plasma state. With this technique, it is possible to analyse multiple elements simultaneously. Up to 70 elements are currently achievable.

5.3.6 Energy-dispersive X-ray spectroscopy (EDX)

With energy-dispersive X-ray spectroscopy (EDX) as per test method ASTM F1375, the atoms in the sample are excited by an electron beam, which causes them to emit X-rays that are characteristic for each element in the sample. This radiation allows the elemental composition of the sample to be measured.

It is important to note that this analysis yields only limited quantitative findings, which in some circumstances can produce results that are not sufficiently precise.

NOTE 1 EDX analysis is often used in conjunction with scanning electron microscopy (SEM, see 5.2.5).

NOTE 2 Quantitative analysis can be heavily influenced by the oxide layer on the surface and possibly do not accurately reflect the entire powder particle.

With this method, only part of a powder particle is analysed rather than the entire sample. This random sampling is insufficient, especially as the chemical composition of individual particles may differ.

Standard test methods are available for determining specific elements in many materials, e.g. ASTM E1409, ASTM E1447, ASTM E1941, ASTM E2371, ASTM E2792, ASTM E539, ASTM E572, ASTM E2465, ASTM E2594 and ASTM E2626.

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[1] 1 ppm = 1 µg/g.

5.4 Characteristic densities

5.4.1 General

The true solid-state density of a material is defined as the ratio of the mass to the volume occupied by that mass.

Apparent and tap densities are useful powder characteristics, enabling the control of different lots, or mixtures of powder.

Apparent and tap densities are relevant in processing of powders as they determine the density of the powder bed after the coating process and during the build, as a compaction of the powder bed occurs due to the weight of the subsequent powder layers.

5.4.2 Apparent density

The apparent density of powders is the ratio of mass to measured volume and is heavily dependent on the particle shape, storage conditions and effect of external mechanical forces. The following methods of determination of apparent density can be used:

a) Scott volumeter method: ISO 3923-2 and ASTM B329 describe the use of a Scott volumeter, which is especially suitable for powders which do not flow freely.

b) Funnel method: The funnel method in accordance with ISO 3923-1 or ASTM B212 and ASTM B417 is suitable for determining the apparent density of metallic powders.

5.4.3 Tap density

The tap density is the powder fill obtained by tapping the powder without additional external pressure until no further volume change is observed. It describes a powder's potential for compressibility.

To determine the appropriate size of graduated cylinder used in tap density measurement, apparent density is measured first.

Tap density is determined using volumetric measurement methods described in ISO 3953 and ASTM B527.

5.4.4 Skeletal (true) density

Apparent and tap density measurements are not true density measurement methods as pores and interparticle voids contribute to the volume. Furthermore, gas atomized powders could contain inclusions of the atomization gas within the individual powder particles. During processing the entrapped gas could escape and form voids or pores in the microstructure of the manufactured component, deteriorating mechanical properties. If the material has no porosity, the true density can be measured by displacement of any fluid in which the solid remains inert. The accuracy of the method is limited by the accuracy with which the fluid volume can be determined.

Skeletal density by gas pycnometry provides the true density of the material as interparticle voids are subtracted during the measurement and can therefore be used to evaluate porosity level within powder particles when compared to nominal density values for the selected material.

NOTE Possibly, pores, cracks or crevices of the powder particles are not completely penetrated by a displaced fluid.

Standard test methods are available for determination of skeletal density, such as ISO 12154 and ASTM B923.

5.4.5 Packing behaviour

Various indices and ratios can be calculated to evaluate the expected packing behaviour. Table 3 provides further information.

Table 3 — Indices, ratios and methods for indication of packing behaviour

Parameter	Equation	Description
Carr index, C	$C = 1 - \rho_b/\rho_{tap}$	Tapping powder in a container after a fixed number of taps.
Hausner ratio	$H = \rho_{tap}/\rho_b$	ρ_b: bulk density
Inter-particle porosity	$I_e = \rho_{tap} - \rho_b \cdot \rho_b/\rho_{tap}$	ρ_{tap}: density after tapping
Volume expansion ratio	V_{drum}/V_{tap}	V_{drum}: volume of powder inside the drum V_{tap}: volume of powder inside container after tapping
$n_{1/2}$	n for which the density is equal to ½ $(\rho_{tap} + \rho_{zero})$	$n_{1/2}$ is a proxy for the rate at which the powder is packing. The lower the $n_{1/2}$, the faster the packing.

5.5 Determination of powder density

5.5.1 Determination of the closed porosity of particles via indirect methods

The closed (internal) porosity of particles has an influence on process stability because the melting behaviour of hollow spheres differs from that of solid spheres. Determining the powder density, i.e., closed porosity of particles, is a means of indirectly measuring the proportion of cavities in powder particles. The following methods are used.

5.5.2 Gas pycnometry

Gas pycnometry is a measurement technique based on the displacement of gas in a calibrated vessel (pycnometer). Gas pycnometry shall be performed in accordance with ISO 12154 or ASTM B923.

5.5.3 Metallographic section with porosity analysis

Powder particles are embedded in a binder and then ground. A camera is used to take micrographs which are then analysed to determine the porosity of the powder particles.

5.6 Shape and morphology

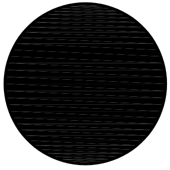
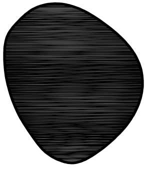
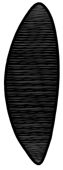
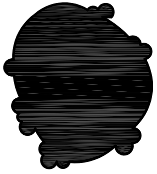
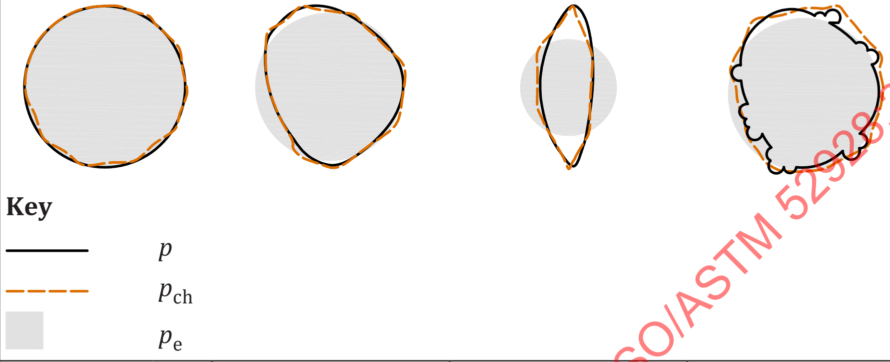
5.6.1 General

The processability of powder, e.g. during the raking/coating step, is strongly influenced by the particle morphology. Typically, spherical particles are desired as they show the best flow behaviour.

The particle morphology is mainly defined by the relative dimensions of the long, intermediate, and short axes of a certain particle. The ratio of intermediate to long diameter and the ratio of short to intermediate diameter is used to define the shape class, see also Table 4.

Smaller particles often adhere to larger particles forming so-called “satellites”. These can be present in virgin powder but typically increase with successive re-use of powder. Satellites emerge during usage by fusion of two or more particles, and in some cases reduce when the fused particles break up into two or more individual particles (see Figure 3).

Table 4 — Particle shape parameters

	Spheroidal	**Nodular**	**Elongated**	**Nodular with satellite**
Particle				
Equivalent morphological parameters				
Circularity[a]	1	<1	≪1	<1
Aspect ratio[b]	1	<1	≪1	Depends on size and location of satellite particles
Convexity[c]	~1	~1	~1	<1

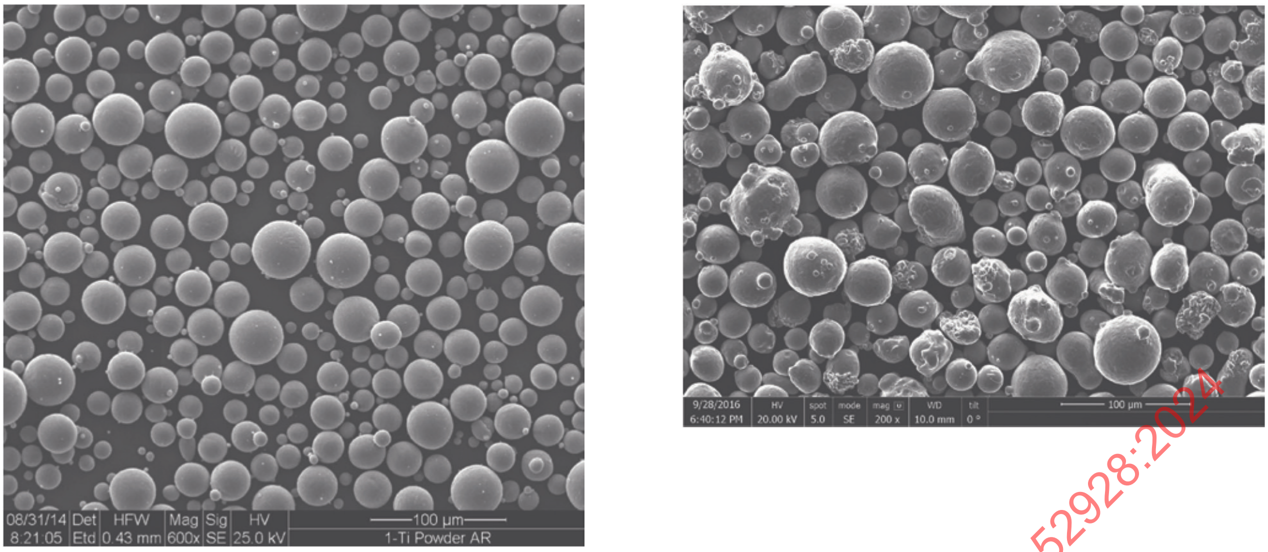
[a] $C = \frac{p}{p_e}$

[b] $A_R = \frac{w}{l}$

[c] $C_x = \frac{p_{ch}}{p}$

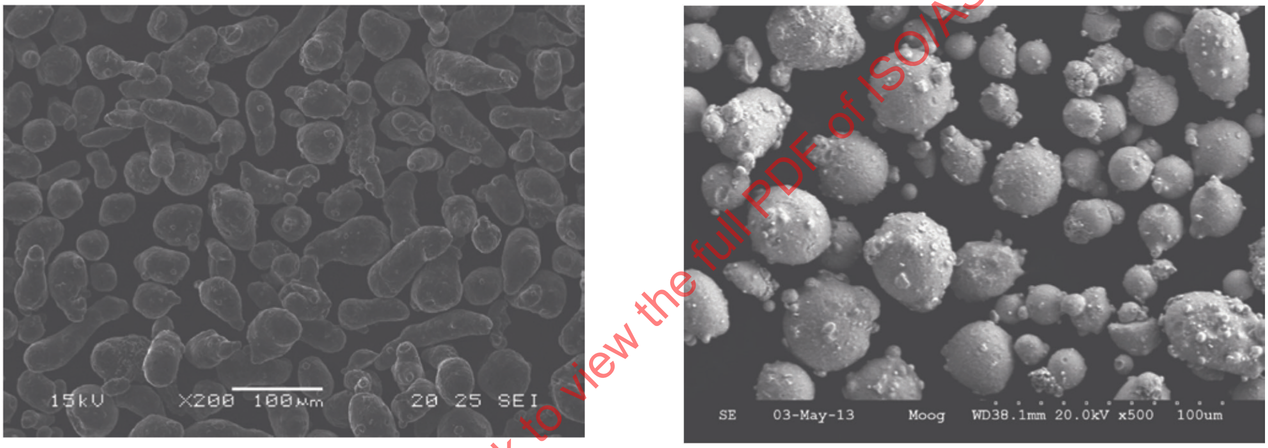
where

A_R is aspect ratio;
C is circularity;
C_x is convexity;
l is particle length;
p is particle perimeter;
p_e is circular equivalent perimeter;
p_{ch} is convex hull perimeter;
w is particle width.



a) **Spheroidal**

b) **Nodular**



c) **Elongated**

d) **Nodular with satellite**

Figure 3 — Particle shapes and satellites

The particle shape has a particular effect on the flow characteristics and coating capacity of the powder.

The following methods may be used to characterize the shape and morphology of particles in a powder sample. Those which provide the greatest amount of information appear first.

5.6.2 Image analysis

Analytical systems such as static image analysis according to ISO 13322-1 or dynamic image analysis according to ISO 13322-2 capture particle shape parameters optically to provide information about deviations from the ideal spherical shape of the particle, for example.

5.6.3 Scanning electron microscopy (SEM) images

Images produced by SEM give a good overview of particle morphology and can be analysed using image analysis systems to generate quantitative statements about the particle morphology of isolated, prepared particles.

5.6.4 Light microscopy images

These images require less preparation, but with a lower resolution than SEM images, they also provide less information.

5.6.5 Determination of specific surface area

Different measuring instruments are used to measure specific surface area via Brunauer, Emmett and Teller theory (BET), see ISO 9277 and ASTM B922.

Determining the specific surface area of powders by gas adsorption in accordance with ISO 9277, a gas, usually nitrogen, is passed through the material under investigation. The amount of gas escaping is measured on the basis of adsorption and desorption processes and is proportional to the total surface area of the powder sample.

This method can be used to indirectly compare powder surfaces. Spherical powders have the smallest surface area.

NOTE There are no or only few reference values available for the powders typically used in additive manufacturing.

5.7 Flowability

5.7.1 General

Flowability is a measure of the ability of a powder to flow, either freely or under an exerted force, e.g. during the raking/recoating step. The requirement of a powder for additive manufacturing to be able to flow is relevant with regard to AM-systems that use piping and/or nozzle to deliver powder, and to mechanical devices to create thin layers necessary to control the manufacturing process.

The amount of cohesion between powder particles quantifies the strength of short-range particle-particle interactions and forces. Where required, this can be measured using powder techniques described in 5.7.5 and 5.7.6.

Powder flowability is a function of multiple factors, particularly the following:

a) cohesive force (e.g. electrostatic charges, Van-der-Waals);

b) powder size distribution (see 5.2) and packing;

c) chemical composition (see 5.3);

d) inter-particles friction, affected mostly by particles' surface roughness and morphology (see 5.6);

e) magnetism;

f) adsorbed water on the particles' surfaces from condensation (see 5.8.1).

As moisture content is a key factor in determining flowability, where drying is required or prohibited by the customer/supplier agreement or the selected flowability standard method (see 5.7.2), this shall be performed in accordance with the conditions specified according to the measurement method, or specified between customer and supplier.

Flowability is not necessarily a reliable indication of spreadability due to other variables that also affect the spreadability, such as type/speed of recoater, layer thickness, etc.

Funnel techniques (see 5.7.2) are primarily used to assess powder flowability with regards to piping/nozzle delivery system requirements. Other techniques, for example rotating drum, may be used to investigate requirements for both flow though piping/nozzle delivery systems and spreadability on the deposition surface.

Where required, spread tests may be performed by the manufacturer to replicate the AM equipment recoating environment to more accurately determine spreadability.

Flowability should be determined to verify

a) the reproducibility of homogenous powder layers, and

b) the transportability of the powder in additive equipment.

The flowability of the powder is influenced by particle size distribution, particle morphology, material density and moisture content.

Due to its significant impact on flowability the moisture content of the powder should be determined (see 5.8.1).

Flow studies are comparable only if the same measurement method is used. The following methods of 5.7.2 to 5.7.7 are suitable for analysing the flowability of metallic powders.

NOTE Currently, the use of a specific method for measuring flowability cannot be definitely recommended.

5.7.2 Determination of flow rate

5.7.2.1 By means of a calibrated funnel in accordance with ISO 4490 or ASTM B213

This method (Hall flowmeter) in accordance with ISO 4490 or ASTM B213 is applicable only to powders which flow freely through the specified test orifice. Powders with particle sizes <100 µm often form bridges in the funnel which falsify the results or make measurement impossible.

5.7.2.2 By means of a calibrated funnel in accordance with ASTM B964

The Carney funnel in accordance with ASTM B964 should be used when powder will not flow through the Hall funnel.

5.7.3 Measuring the angle of repose

Measuring the angle of repose in accordance with ISO 4324 provides information about the pourability of powders.

5.7.4 Ring shear test method

The ring shear test method in accordance with ASTM D6773 is used to measure the yield strength and flow properties of a powder.

5.7.5 Rotating drum with dynamic image analysis

This method measures the flow properties of powders by automatically analysing images of a powder sample as it avalanches in a rotating drum.

5.7.6 Powder rotational rheometer

The powder rheometer measures the flowability of powders by moving a rotating screw blade at constant pressure through powder samples prepared to standard parameters. The flowability of the powder is characterized by the rotational and vertical resistance of the blade (torque and force).

5.7.7 Hausner ratio (ratio of tapped to bulk density)

The Hausner ratio is used to measure flowability in conventional powder metallurgy (sintering and compaction) and does not provide a clear statement about the suitability of powders for additive manufacturing processes. There is no correlation with the flow measurements obtained from the other methods listed here for the powders typically used in additive manufacturing, which are much finer than those associated with conventional powder metallurgy. This does not mean that packing behaviour is not relevant to characterize AM processes, especially the impact of fines or the presence of humidity, which can change the packing behaviour (see 5.4.5).

5.8 Contamination

Contaminations are undesirable elements that can be present in the virgin feedstock or picked up during usage. Usually contaminations adversely affect the powder properties and shall be prevented and/or minimised. As agreed by customer/supplier, the powder batch used shall not include foreign material containing morphology, chemistry or impurities outside of specification. Possible contaminants and related effects are listed in the following subclauses.

5.8.1 Moisture content

5.8.1.1 General

The moisture content describes the amount of water the powder sample holds. Moisture can be absorbed from the ambient air or from insufficiently dried containers or tools.

Moisture in the powder can have the following effects:

a) it is a potential source for hydrogen and oxygen into the final part;

b) it impairs the flow behaviour (see 5.7 for effects);

c) depending on the ambient temperature, condensation of water on surfaces such as the laser entrance window or sensors can have a sensitivity effect on the process.

5.8.1.2 Moisture analysis

5.8.1.2.1 General

The moisture content of a powder has a significant influence on its flow characteristics, as well as the uptake of oxygen and hydrogen by the powder. In the event of inadequate monitoring of the operating conditions, or after incidents of possible powder contamination, it should be regularly tested, using one of the methods in ASTM F3606 and described in 5.8.1.2.2 to 5.8.1.2.4.

5.8.1.2.2 Thermogravimetric analysis (TGA)/Loss on drying

Thermogravimetric methods are weighing-drying methods which involve drying samples to constant mass. The change in mass is interpreted as released moisture.

NOTE Loss on drying techniques report the evaporation of other volatile organic substances that are conflated with the evaporation of water when reporting powder moisture. Caution should be exercised when reporting moisture content via loss on drying techniques without appreciating the powder lifecycle history and potential exposure to other liquid agents.

The suitability of this method should be checked for the expected moisture content, as it has been found to be less sensitive than the methods described in 5.8.1.2.3 to 5.8.1.2.4.

5.8.1.2.3 Oven desorption followed by Karl Fischer titration

Karl Fischer titration (determination of H_2 and back calculation to moisture content) is a quantitative chemical method for determining water content which is based on the oxidation of Sulphur dioxide by iodine in a methanolic alkaline solution. Since this reaction consumes water, it can only continue for as long as water is still present in the analyte. Once all the water has been consumed, the reduction of iodine ceases. The resulting brown discoloration provides a visual indication of the endpoint. Titration may be volumetric (simple apparatus) or coulometric (requires special reagents).

5.8.1.2.4 Electronic moisture meters

Commercially available electronic moisture meters determine moisture content by measuring electrical resistance between two electrodes, by measuring the capacitance of an electric capacitor, or using microwave methods.

NOTE Comparative residual moisture can be rapidly analysed using relative humidity measurements.

5.8.2 Impurities

Impurities are traces of elements present in small quantities within the powder that cannot be entirely avoided in the manufacturing process. The metal powder shall be free from detrimental amounts of inclusions and impurities.

NOTE Usually a maximum allowable value (in at% or *m*%) is specified depending upon the requirements of the manufacturer and/or customer, depending on the application of the part being produced.

5.8.3 O/H content

During their life cycle, metal powders tend to pick up oxygen and hydrogen via formation of surface oxides, hydroxides as well as an adsorbed film of moisture. Both hydroxides and the moisture film can form oxides upon heating. The relative amount of oxygen and hydrogen within a powder feedstock shall be specified to assure certain properties in the final part. If specified maximum values are exceeded, material properties can change due to either internal/external oxidation (O) or embrittlement (H).

NOTE Embrittlement as a result of H requires H to be diffusible. No evidence yet to suggest that laser beam melting leaves H in a diffusible form.

5.8.4 N content

In processes using nitrogen as the inert atmosphere, nitrogen can increase in solution during melt and consolidation across a variety of AM processes. This can lead to the formation of greater concentrations of deleterious nitrides and could chemically suppress the formation of desired phases or result in increased tensile strength due to interstitial strengthening.

5.9 Absorption rate of the powder

5.9.1 General

Since infrared lasers are usually used in laser beam melting and energy is supplied to the powder by absorption of the laser beam, the absorption rate of the powder for the wavelength of the laser radiation has a significant influence on the process.

The absorption rate is determined by means of a spectrogram in which the absorption value is selected according to the wavelength of the laser used. Powders with lower absorption rates require higher laser power and/or longer exposure times to melt.

5.9.2 Diffuse reflectance infrared Fourier transform (DRIFTS)

This method of infrared spectroscopy may be used on unprepared powder samples and measures both the reflection and absorption of radiation.

6 Powder life cycle

6.1 Batch requirement

6.1.1 General

The following clauses cover meaning of terms specific to powder batch management and traceability. Specifically it covers the meaning of a batch and designation of powder volumes which are the product of powder handling activities.

6.1.2 Specification

A user can define different specifications for purchasing powder (virgin powder specification) and for reusing powder (i.e used powder specification). The specification of the virgin powder can differ from the specification of the used powder, for example by being more restrictive.

6.1.3 Batch

One batch may be used in several builds with different process parameters. However, process parameters can influence the aging of the feedstock in which case this consideration should be evaluated.

6.1.4 Blend

The specific requirements for a powder blend are typically determined by the application or by agreement between the supplier and end-user. A distinction is made between blended powders and mixed powders, in which case blended powders are combinations of powders with nominally identical composition, whereas mixed powders are combinations of powders with different specifications such as composition, morphology, etc. The product of a blend may be analysed to ensure powder properties meet desired specification, and may be designated as new batch.

6.1.5 Powder mix

Mixed powders should not be used for production, due to potential variance in material properties. The product of a mix shall not be designated as a new single batch. Mixed powders may be allowed where feedstock repeatability and reuse studies have been established and results of the study show compliance with the powder specification.

6.1.6 Combine

Considerations should be taken when combining powders of markedly different reuse age/iteration due to expected differences in powder properties. The product of a combine can be designated as a new individual batch but its history shall be recorded and traced.

6.1.7 Reuse metric

If an aspect of a build job run is not fixed (e.g. differing build layouts or process parameters in successive builds) then it is recommended to apply a continuous scale for reuse (e.g. total exposure time), as powder degradation rates are sensitive to process inputs and therefore will vary depending on build parameters, layouts and part geometry.

If all aspects of a build job run are fixed, then use of an iterative metric (e.g. build number) is applicable.

When two or more powder batches are blended, the resultant batch will retain the highest reuse metric from the constituent batches. For example, if a used batch of reuse metric “3 builds” is blended with a used batch of “2 builds” and a virgin batch (“0 builds”), the resultant batch will have an effective reuse metric of 3 builds.

NOTE Various industry associations such as SAE International provide recommended practices to measure, track and characterize the history of powder feedstock for specific applications, e.g. ARP7044 “Powder History Metric and Labeling Schema” for use in aerospace.

6.2 Traceability

6.2.1 General

The user shall operate a system by which every event – an action or process – that a powder batch is subjected to, and any information or data associated with said event, is tracked and recorded. The system shall allow for realization of a powder batch event history from current state back to original receipt of powder at user location.

Anything that can help in the monitoring and traceability based on the validated method is advised. More general information on the traceability can be found in ISO/ASTM 52920 for quality requirements of AM sites.

6.2.2 Event history

The system should capture the following actions and processes as a minimum:

a) split of powder batch to form one or more sub-batches;

b) combine of a powder batch with another batch or sub-batch;

c) blend of a powder batch with another batch or sub-batch;

d) sieve of a powder batch;

e) loading of a batch or sub-batch into an AM system;

f) processing of a batch in an AM system;

g) unloading of a batch or sub-batch from an AM system;

h) sampling of a powder batch.

6.2.3 Powder state

The following information for a powder batch at any given time should be known and recorded:

a) location on site (e.g. storage area, quarantine, production floor, loaded in an AM system);

b) data from original batch certificate of conformance (as provided by supplier);

c) action and process history;

d) quantity remaining;

e) reuse metric.

6.2.4 Labelling

In order to ensure traceability throughout the powder life cycle at the user location, powder containers shall have a unique label or code which either contains the below information or from which the below information can be retrieved (e.g. barcode linked to a digital database) in addition to that stipulated by ISO/ASTM 52907:

a) metal or alloy designation (e.g. Nickel alloy 718);

b) batch number/unique identifier;

c) nominal particle size range (e.g. 15 μm to 45 μm);

d) delivery date/date of creation;

e) net mass of contents;

f) tare mass of the packaging;

g) powder state.

6.3 Handling

6.3.1 General

Any handling operation should always take place under stable environmental conditions. It is recommended to monitor temperature and humidity during all handling operations so that significant shifts can be identified and operations managed accordingly. The user shall determine limits for temperature and relative humidity within their facility, when handling powders.

NOTE Typical limits are 15 °C to 25 °C and RH below 55 % depending on material, process, and application.

Reactive metals and alloys such as titanium and copper which are prone to oxidation in ambient conditions should be handled as much as possible under an inert shielding gas e.g. Argon. This can be achieved by performing handling operations within a glove box operating with an inert gas atmosphere, or by operating a closed-loop powder handling system (physical connections between machines, containers or autonomous modules whereby neither powder is exposed to atmosphere nor operators are exposed to powder) where the carrier gas is an inert gas.

6.3.2 Storage

Ideally, powder should be stored in the same conditions as production or test environment in order to minimize risk of moisture ingress (condensation) upon transfer. Specifically users should avoid storing powder in temperatures less than 15 °C, or in storage locations with unstable temperature and humidity levels. Shelf life of sealed containers from the supplier will be stipulated by the supplier, but will be dependent on alloy-base and storage conditions. Powder may be packed under an protective inert atmosphere, e.g. Argon, to minimize oxidation and moisture ingress. Such practice is recommended for reactive alloy-base powders. Containers with integrated sensors (local or IoT) for temperature, humidity and oxygen may be used to aid in monitoring and control of the environment within the container and with respect to external environment. Such systems may be used to alert users if undesirable conditions are reached.

If a powder has been in continuous storage for 6 months or longer, it is advised to establish whether key properties such as oxygen content, moisture and flow are still within specification due to possible impact of long term exposure. Alternatively a validation build may be used to establish whether target part properties can be achieved.

6.3.3 Transfer

It is recommended to transfer powder in a closed-loop system where possible to minimise contamination with the atmosphere or foreign materials, and minimise contact of operators with powder. During transfer, the powder and the container ideally should be at the same temperature – again to avoid condensation upon transfer. All components of the transfer should be normalised in the same environment prior to conducting the operation. Alternatively, a system of sensors (local or IoT, i.e. “Internet of things”) for humidity, temperature and oxygen on the AM and powder handling equipment and external environment may be used to determine that a stable and safe condition exists prior to transfer of powder.

Powder transfer equipment should be designated for a single alloy-base or suitably cleaned to avoid cross-contamination. Methods for cleaning shall be conducted to a fixed internal operating procedure or external standard and validated to confirm effectiveness and may be subject to agreement between customer and manufacturer.

6.3.4 Repacking

Full traceability (event tracking and labelling) shall be maintained if the powder is transferred from one container to another e.g. from small containers to a single larger container. Reusable containers, such as metal hoppers, should be designated for use with a single alloy-base in order to minimize impact of cross contamination, and should be thoroughly cleaned between transfer of different batches. Again, cleaning should be conducted to a fixed and validated method.

6.4 Recycling/reuse of feedstock

Blends shall be homogenised to minimise the process variations (e.g. virgin with used). The percentage of virgin and used powder may be related to mechanical properties of the final part, such as an acceptable loss of strength. Some materials tend to provide higher strength (e.g. titanium) because of the oxygen or nitrogen increase along its reuse.

For example, ASTM F 3001 for Ti64-ELI requires that the ratio of virgin and used powder shall be recorded if applied. The maximum recycling iterations shall be recorded and validated.

The maximum reuse metric for a given production run may be determined by the following and using qualification data obtained by the manufacturer for a set of fixed processing steps:

a) set maximum number of builds;

b) set maximum amount of exposure time or total exposure energy;

c) final build with build parts test coupons;

d) final build powder samples properties against predefined specifications.

Different reuse methods may be used in production and should be documented and validated (e.g. sieving method, mesh size, amount of powder after sieving, etc.). The list below gathers different types of methods that may be used and have been proven to work efficiently:

a) no refreshing with virgin powder:
    1) no reuse;
    2) discrete reuse until no more powder is available;
    3) continuous reuse by topping up the powder directly after production;
    4) in-process powder reuse by topping up the powder during production;

b) refreshing with virgin powder:
    1) refreshing with virgin powder after every build;
    2) continuous reuse by topping up the used powder and adding virgin powder when required.

6.5 Disposal

Any powder that reached the maximum reuse metric shall be labelled as mix for prototyping or disposed as per local authority regulations or third party for recycling.

7 Powder quality assurance

7.1 Documentation requirements

The powder supplier generally provides the user with the following information about the powder characteristics:

a) chemical composition;

b) particle size distribution;

c) flow characteristics;

d) tap density;

e) bulk density;

f) packaging.

Individual arrangements may be made between the powder supplier and user.

The documentation indicates how each quality characteristic has been determined (see Clause 5). Unless otherwise agreed between supplier and customer a "Type 3.1" certificate of compliance can be issued in accordance with EN 10204.

7.2 Certificate of analysis (CoA)

Where required by a purchase specification, a certificate of analysis or factory test certificate should contain batch-specific measurements of powder parameters (characteristic values) that can have a critical impact on the manufacturing process. The factory test certificate may also contain parameters which are extremely difficult or impossible to verify during the incoming goods inspection.

Each factory test certificate should include the following data:

The particle size distribution has a significant influence on flowability and bulk density and thus contributes to the processability and part characteristics. Typical data are the D_{V10}, D_{V50} and D_{V90} values. These correspond to the particle size at which 10 %, 50 % or 90 % of the volume fraction of the powder is smaller than this value. A volume- or mass-related analysis is preferable to a numerical analysis since small particles account for only a small mass or volume fraction, even when they are present in large numbers.

A large number of large particles impair the surface quality and fine detailing (detail resolution) of the part. The D_{V90} value can be used to indicate the coarse fraction; i.e. 10 % of the volume fraction of the powder is larger than this particle size.

A high proportion of fine particles produces more fine particulate matter which can contaminate the AM machine and its surroundings. The smaller the particles, the larger the surface-to-volume ratio and the stronger the surface forces and electrostatic charge. This impairs flowability and can cause powder deposits to accumulate on the recoating system, for example. The D_{V10} value serves as a measure of the fine fraction; i.e. 10 % of the volume fraction of the powder is smaller than this particle size.

D_{V10}, D_{V50} and D_{V90} values shall be indicated as a minimum. It is advantageous to indicate the particle size distribution in full.

7.3 Sampling

7.3.1 General remarks

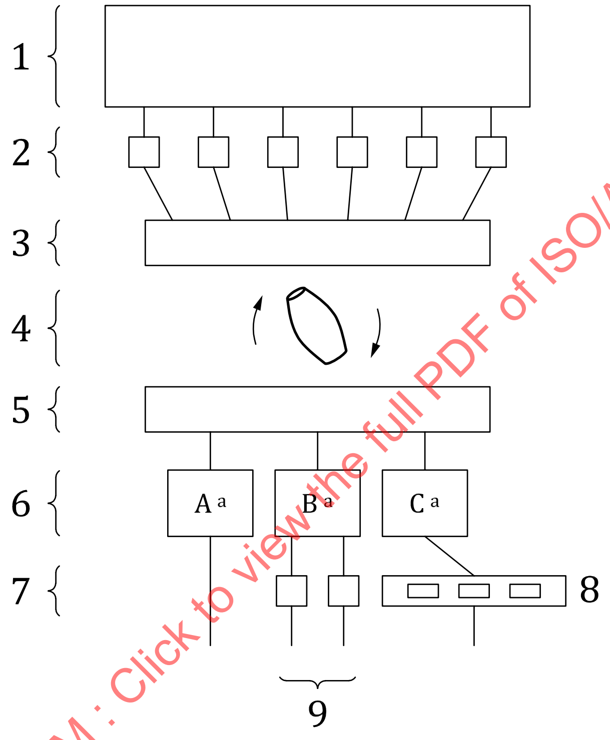
It is recommended to conduct sampling either in accordance with ISO 3954 or ASTM B215.

These standards describe in detail numerous sampling methods for obtaining a small quantity of material that is truly representative of the larger basic population of powder tested (see, for example, Figure 4).

They also describe procedures for dividing powder samples into smaller test samples for the purpose of determining the chemical, physical, and mechanical properties of the material. Here, a distinction is made between sampling moving powders (for example in-line or when being transferred from one container to another) and static powders that have already been packaged for transport. Furthermore, these standards explain how to sample composite materials containing binders or additives.

It is generally advisable to collect powder samples from moving/free-flowing volumes of powder, taking care to ensure that the sample is representative of the entire volume of powder being tested.

If samples are collected for test methods which depend on the moisture content of the powder, e.g. to determine flowability (see 5.8), precautions shall be taken to avoid altering the moisture content of the powder samples in the time between collecting the sample and performing the test. Because the powder is highly reactive to ambient air humidity, it is particularly important to minimize the exposure time to changed ambient climatic conditions and to use suitable powder sampling containers.



Key

1	lot
2	increments
3	gross sample
4	(blending)
5	composite sample
6	test samples
7	portion
8	test pieces
9	(determination)
a	Property.

Figure 4 — Sampling scheme in accordance with ISO 3954

7.3.2 Characterization of virgin powder and powder blends

It is advisable to test not only each new batch of virgin powder, but also each powder mix comprising used and virgin powder in accordance with the measurement methods listed in this document. This is especially