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Procesi aditivne proizvodnje - Lasersko sintranje polimernih delov/laserska fuzija plasti polimernih delov - Kvalifikacija materialov (ISO/ASTM/DIS 52925:2020)

Additive manufacturing processes - Laser sintering of polymer parts/laser-based powder bed fusion of polymer parts - Qualification of materials (ISO/ASTM/DIS 52925:2020)

Additive Fertigung - Lasersintern von Polymerteilen/laserbasiertes pulverbettbasiertes Schmelzen von Polymerteilen - Qualifizierung von Materialien (ISO/ASTM/DIS 52925:2020)

Procédés de fabrication additive - Frittage laser de pièces polymères / fusion laser sur lit de poudre de pièces polymères - Qualification des matériaux (ISO/ASTM/DIS 52925:2020)

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Contents

Page

Foreword ..... **iv**

1 Scope **1**

2 Normative references **1**

3 Terms and definitions **1**

4 Symbols and abbreviations **1**
4.1 Symbols 1
4.2 Abbreviations 2

5 Sampling **2**
5.1 General 2
5.2 Characterisation of new (virgin) powder and powder mixes 2
5.3 Characterisation of used powder 2

6 Factory test certificate **3**
6.1 General 3
6.2 Particle size distribution 3
6.3 Residual monomer content/extract content 3
6.4 Supplementary data 3

7 Factors influencing processability **4**
7.1 General 4
7.2 Flowability of the powder 4
7.3 Relative humidity of the powder (surface moisture) 4
7.4 Particle size distribution 5

8 Factors affecting part quality **5**
8.1 General 5
8.2 Melting behaviour, melt flow and MVR 6
8.2.1 General 6
8.2.2 Laboratory methods 6
8.2.3 Melt volume-flow rate (MVR) 6
8.3 Melting temperature and recrystallisation temperature 7

Annex A (informative) **Hausner ratio (H_R)** **9**

Annex B (informative) **Determination of the melt volume-flow rate (MVR)** **12**

Annex C (informative) **Round robin MVR test** **15**

Bibliography **18**

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).

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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.

The committee responsible for this document is 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.

This is the first edition of this document.

Additive manufacturing processes — Laser sintering of polymer parts/laser-based powder bed fusion of polymer parts — Qualification of materials

1 Scope

This document establishes specific parameters and recommendations for the qualification of polymeric materials intended for laser sintering. The parameters and recommendations presented in this document relate mainly to the material polyamide 12 (PA12), but references are also made to polyamide 11 (PA11). The parameters and recommendations set forth herein may not be applicable to other polymeric materials.

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 ASTM 52900, *Additive manufacturing — General principles — Terminology*

ASTM D6779*Standard Classification System for and Basis of Specification for Polyamide Molding and Extrusion Materials (PA)*

3 Terms and definitions

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

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

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

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

4 Symbols and abbreviations

4.1 Symbols

The following symbols are used throughout this standard:

Symbols	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_R	Hausner ratio	—
s_r	standard deviation of repeatability	—
s_R	standard deviation of reproducibility	—
T_B	processing temperature range	°C
T_{ic}	initial crystallisation temperature	°C
T_{im}	initial melting temperature	°C

Symbols	Designation	Unit
V_∞	bulk volume	mL
V_0	tapped volume	mL
η_{rel}	relative viscosity	–
ρ_∞	bulk density	g/mL
ρ_0	tapped density	g/mL

4.2 Abbreviations

The following abbreviations are used throughout this standard:

AM additive manufacturing

DSC dynamic scanning calorimetry

GPC gel permeation chromatography

MFI melt flow index

MFR melt mass flow rate

MVR melt volume flow rate

PA11 polyamide 11

PA12 polyamide 12

RoHS restriction of the use of certain hazardous substances

5 Sampling

5.1 General

When analysing a small powder sample to determine its quality and suitability for laser sintering, it must be ensured, that this sample is representative of the powder as a whole.

5.2 Characterisation of new (virgin) powder and powder mixes

Each new batch of virgin powder shall be tested and also each powder mix comprising used and virgin powder should be tested in accordance with the measurement methods listed in this standard. Test of powder mix is recommended for series serial production to ensure consistent part quality. To minimise the scope of testing and ensure a high level of powder homogeneity, batch size of mixed powder should be as large as possible.

5.3 Characterisation of used powder

Since different temperature histories within the used powder of a part cake can lead to significant differences in material quality, the total quantity of powder shall be homogenised to obtain a representative powder sample.

In practice this can be achieved by thoroughly mixing the powder in a mixer, for example. Once mixed, the sample can be taken from any part of the powder. If a sufficiently large mixer is not available, several samples (up to five, each weighing 20 g) may be taken from different areas and mixed together. Samples for analysis may then be taken from this mix.

IMPORTANT — Ideally, the total volume of powder should be homogenised rather than individual samples.

6 Factory test certificate

6.1 General

The factory test certificate should contain batch-specific measurements of powder parameters (characteristic values) that can have a critical impact on the manufacturing process.

Each factory test certificate shall include the data according 6.2 to 6.4.

6.2 Particle size distribution

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 impairs 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 are. 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.

The median particle size (D_{v50} value) provides a good indication of the resulting surface roughness of parts since this is largely determined by particles adhering to the parts or partially fused particles.

IMPORTANT — 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.

7.4 explains how to determine the particle size distribution.

6.3 Residual monomer content/extract content

The residual monomer content/extract content of a laser sinter powder should be kept to a minimum level to minimise the release of gases from the powder. These monomeric gas releases can condense on colder areas of the additive manufacturing machine and soil the system as a result. Optical elements soiled in this way absorb laser radiation, leading to a reduction in the laser power delivered to the build field. This can have a detrimental effect on the mechanical properties and the part density. Soiling of mechanically stressed components can accelerate ageing or in extreme cases, cause them to fail.

IMPORTANT — The residual monomer content is indicated as percentage mass and should be below 0,5 % for polyamide 11 and polyamide 12 powders.

6.4 Supplementary data

Normally, the powder formulation is confidential and is not disclosed. However, the purchaser can generally assume that the material supplier keeps to his formulation and that the content of fillers and additives remains constant from one batch to another and is monitored during production. No change to the formulation or to the additives is acceptable without prior agreement from the customer It is unusual to explicitly specify the formulation in the factory test certificate, as is also the case with

plastic pellets for injection moulding applications. Powder shall be certified to specification call-out such as ASTM D6779. Appropriate certificates (e.g. Safety Data Sheet, "Globally Harmonized System of Classification and Labelling of Chemicals", EU Directive 2011/65/EU) are issued to confirm that the formulation contains no harmful substances. Important components and their effect on part characteristics are listed below:

— stabilisers: these prevent thermal degradation within the polymer and also influence the mechanical characteristics of the resulting part.

— fillers (e.g. glass beads): fillers in the polymer influence the mechanical characteristics of the part.

— flame retardants: these influence the flammability of the parts.

— additives: certain additives can be contractually excluded, depending on subsequent use.

IMPORTANT — The supplementary information shall be agreed between the contractual partners, depending on the desired area of application.

7 Factors influencing processability

7.1 General

Many characteristics of the polymer powder ultimately determine whether or not a part produced by laser sintering conforms to requirements. However, the tolerance limits of many of these characteristics largely depend on the AM machine and/or the processing parameters used. For this reason, this section deals only with those factors which make processing fundamentally unfeasible; in other words, the measurements described below can be used to exclude unsuitable material.

7.2 Flowability of the powder

The flowability and flow characteristics of the powder determine the quality of the powder recoating. If a powder is not free-flowing because the particle shape and/or distribution is unfavourable or its electrostatic charge is too strong, neither the powder feed to the process chamber nor the application of a thin powder layer in the AM machine can be guaranteed. Any powder with poor flow characteristics shall be detected and excluded. . There are various possibilities to determine the flowability of powders and their use for laser sintering powders is under investigation. One possibility that has shown good results in a round robin test (Annex A) is the determination of the Hausner ratio H_R. This is based on determination of flowability as per ISO 6186 and determination of bulk density as per ISO 60. Whilst the last two methods are defined very precisely in the corresponding standards, determination of the Hausner ratio has yet to be standardised. Annex A of this standard describes in detail the definition of the Hausner ratio H_R, its determination by measurement and its significance.

7.3 Relative humidity of the powder (surface moisture)

When grains of powder are in motion, the surfaces of individual particles come into contact with one another and then separate again, leading to charge separation which causes the powder to develop electrostatic charge. Since a polymer is an insulator (non-conductive material), no charge equalisation can occur within the individual particles. Discharge takes place across the surface moisture of the particle, as well as via the ambient air. The electrical conductivity of the powder generated by the surface moisture is therefore critical for breaking down electrostatic charges inside the powder volume.

Since it depends on the moisture at the particle surface, measurement of the moisture using moisture scales is not informative. With this method, the moisture loss of a sample is measured by increasing the temperature. However, in the case of polyamides, the polymer itself also absorbs water and this moisture also escapes on heating. Thus it is not possible to distinguish whether the water played an active role in discharge at the particle surface or was trapped inside the particles.

A moisture meter with probe is therefore a suitable alternative. This measurement method determines the relative humidity of the air. The probe can be inserted in the powder since the relative humidity of the air is an indirect indication of the surface moisture of the powder. The higher the measured relative humidity, the higher the surface moisture of the powder. To obtain reproducible measurements, the probe should be inserted to the same depth in the powder bed each time. The depth should be a minimum of 200 mm. For PA12 and PA11, relative humidities of 40 % to 60 % measured in this way have proved ideal for processing the powder.

7.4 Particle size distribution

As already described in 6.2, particle size distribution has an influence on processability and/or part quality and for this reason it should also be monitored in powder mixes comprising used and virgin powder. Problems with processability can occur if the coarse fraction is too high. These large particles can cause defects during recoating (e.g. stripes or scratches) or even displace small parts in extreme cases. The coarse fraction also has an influence on the surface quality and detail resolution of the part. The coarse fraction is characterised by the D_{v90} value.

The fine fraction also causes defects during coating due to interparticle interactions, electrostatic charging and resulting poor flowability. The fine fraction is characterised by the D_{v10} value.

Various measurement methods are available to determine the coarse and fine fraction by means of analysis.

The best-known is laser diffraction pursuant to ISO 13320. Here the powder, dispersed in compressed air (dry measurement) or in a liquid dispersant (wet measurement) is placed in the path of a laser beam and the particle size distribution is calculated using detectors which measure the light scattering pattern.

Optical image analysis is another method. A distinction is made between static image analysis for resting particles (ISO 13322-1) and dynamic image analysis for particles in motion, dispersed in air or liquid (ISO 13322-2). Both versions are based on the analysis of 2D projected images of powder particles, which provide information about particle size distribution and particle shape (including sphericity).

Sieving analysis (ASTM D1921 or ISO 3310) is a robust method of analysing powders. Here a powder sample is poured into a tower containing several sieves. The top sieve has the largest mesh width and each lower sieve has a smaller mesh width than the one above. By weighing the individual fractions retained in each sieve it is possible to determine the particle size distribution of the powder by weight of the single fractions.

For powder monitoring purposes, a simplified version comprising just one coarse sieve could be used to determine purely the coarse fraction and establish an internal threshold for coarse material. The mesh width of the coarse sieve should correlate with the layer thickness subsequently required (the thinner the layers, the finer the sieve). Mesh widths from 120 µm to 200 µm shall be used. The coarse fraction retained on the sieve should not exceed 5 %. No contaminants or extremely large particles/ agglomerates should be visible. This method is a very reliable means of detecting insufficiently sieved powder or a sieve damage during powder processing.

8 Factors affecting part quality

8.1 General

A range of factors have an impact on part quality. Material characteristics which have an influence on the finished part and can be measured using analytical methods are described in the following sections.