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Additive manufacturing of polymers — Feedstock materials — Qualification of materials for laser-based powder bed fusion of parts

Fabrication additive de polymères — Matières premières — Qualification des matériaux pour la fusion laser de pièces sur lit de 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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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 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).

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INTERNATIONAL STANDARD **ISO/ASTM 52925:2022(E)**

Additive manufacturing of polymers — Feedstock materials — Qualification of materials for laser-based powder bed fusion of parts

1 Scope

This document provides guidance and recommendations for the qualification of polymeric materials intended for laser-based powder bed fusion of polymers (PBF-LB/P). 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 cannot 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 — Fundamentals and vocabulary*

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:

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

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

4 Symbols and abbreviations

4.1 Symbols

The following symbols are used throughout this document:

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
V_∞	bulk volume	mL
V_0	tapped volume	mL

Symbols	Designation	Unit
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 document:

DSC	differential 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 PBF-LB/P, it shall be ensured that this sample is representative of the powder as a whole.

5.2 Characterisation of virgin powder and powder blends

Each new batch of virgin powder shall be tested and also each powder blend comprising used and virgin powder should be tested in accordance with the measurement methods listed in this document. Test of powder blend 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 blended 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 blending the powder in a mixer, for example. Once blended, the sample can be taken from any part of the powder. If a sufficiently large mixer is not available, several samples (at least five, each with a mass of at least 20 g) may be taken from different areas and blended together. Samples for analysis may then be taken from this blend.

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

6 Factory test report

6.1 General

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

Each factory test report should include the data according to 6.2 to 6.4.

6.2 Particle size distribution

The particle size distribution has a significant influence on spreadability 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 spreadability and can cause powder deposits to accumulate on the powder spreading device, 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.

Powder supplier and user have to agree on the method to measure the values and the tolerances. D_{V10}, D_{V50} and D_{V90} are mandatory and depending on the PSD (e.g. bimodal) there should be more data in the report or the total analysis (graph and data) of the PSD.

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.

Determine the particle size distribution according to 7.4.

6.3 Residual monomer content/extract content

The residual monomer content/extract content of a PBF-LB/P 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.

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 report, as is also the case with plastic pellets for injection moulding applications. It is recommended that powder be certified to specification call-out such as ASTM D6779[16]. 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,

— flow agents: these enable less restricted movement of particles relative to one another,

— fillers (e.g. glass beads, fibres, ceramic particles, impact modifiers): 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 is recommended to 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 PBF-LB/P 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 subclause 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 Spreadability of the powder

The spreadability and flow characteristics of the powder determine the quality of the powder for the powder spreading device of a PBF-LB/P system.

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 PBF-LB/P powders is under investigation. One possibility that has shown good results in a round robin test (see Annex A) is the determination of the Hausner ratio H_R. This is based on determination of pourability 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 document describes in detail the definition of the Hausner ratio H_R, its determination by measurement and its significance.

NOTE The Hausner Ratio is recommended, since there is a good relation to spreadability of powders in PBF-LB/P.

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 blends comprising used and virgin powder. Problems with processability can occur if the coarse fraction is too high. These large particles can cause defects during powder spreading (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 spreadability. 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.

One method 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[14] 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 no analysis of the particle size distribution is necessary. 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 subclauses.

8.2 Melting behaviour, melt flow and MVR

8.2.1 General

The flow characteristics of a polymer melt are largely determined by the molecular mass distribution and the temperature, pressure and shear rate of the melt. The higher the melt temperature, the lower the viscosity. This effect is even more pronounced with PBF-LB/P than with conventional polymer processing methods, since shear and pressure are not involved. In this case, temperature, molecular shape and chain length determine whether the powder forms an adequate melt film, how many pores remain in the part and how well the layer bonds to the previous layer. The flow characteristics of the melt thus give an indication of the part mechanics and the layer-to-layer bonding.

The process parameters (e.g. laser power and scan speed) are normally selected to produce a homogenous melt layer. Too low melt flow leads to the formation of pores in the component or poor layer bonding. Both outcomes can have an adverse effect on part mechanics. Thus, it is extremely important to control the melt viscosity of both virgin powders and powder blends in order to produce high-quality parts.

8.2.2 Laboratory methods

The viscosity is determined at molecular level via the molecular structure and molecular mass distribution. GPC (gel permeation chromatography) can be used to obtain information about the molecular chain length and the static distribution of chain lengths in the material. However, since the method is extremely time-consuming and costly, it is recommended only for detailed analyses.

Determination of the solution viscosity also provides information about the average chain length and molecular mass. This is done using polymers in varying concentrations dissolved in specific solvents. Typical solvents include *m*-cresol, tetrachloroethane and concentrated sulphuric acid. Polyamides are measured in a capillary viscometer in accordance with ISO 307. The relative viscosity, η_{rel}, is calculated from the ratio of flow time of the polymer solution to that of the pure solvent.

$$\eta_{\mathrm{rel}} = \frac{t_{\mathrm{f,pol,sol}}}{t_{\mathrm{solv}}} = \frac{\eta_{\mathrm{pol,sol}}}{\eta_{\mathrm{solv}}} \qquad (1)$$

The greater the η_{rel} value within a polymer class, the greater the average molecular mass and the higher the viscosity of the corresponding polymer melt. Since the determination of solution viscosity requires very complex apparatus and the use of highly toxic solvents, it is advisable to perform this measurement only in a suitably equipped test laboratory as part of production control.

The two above mentioned methods are suitable for in-process quality control of the PBF-LB/P powder to a limited extent.

8.2.3 Melt volume-flow rate (MVR)

8.2.3.1 General

Measurement of the melt volume-flow rate (MVR) is a suitable means of characterising the viscosity of the polymer melt at a specified applied weight and a specified temperature. The melt mass-flow rate (MFR) or the melt flow index (MFI) are also widely used.

Determination of the MVR pursuant to ISO 1133-1 and ISO 1133-2 is done by means of a capillary rheometer, whereby the material (granules or powder) is melted in a heated cylinder and forced through a defined orifice (capillary) by the pressure generated by the applied weight. The volume of polymer melt extruded – the extrudate – is determined as a function of time. Since the measurement setup is relatively simple, this process is suitable for in-process monitoring of material in production facilities.

When measuring polyamide, the moisture content of the sample is critical. Since polyamides can absorb water and, especially in the case of powders, moisture can accumulate on the (large) specific surface area of the powder, due to the small powder particles, identical ambient conditions and sample preparation methods are essential to ensure reliable measurements. Furthermore, the molecular mass of the sample can change during measurement because the polymers can both break down and form within the melt. It follows that measurements are comparable only if the same measurement cycles are followed, e.g. fixed preheating times.

8.2.3.2 MVR standard operating procedure

B.5 contains a sample standard operating procedure for determining the MVR value when using PA12 powder.

8.2.3.3 Round robin MVR test

The results of a round robin test (interlaboratory test) to determine the MVR value are presented in Annex C.

8.2.3.4 Powder monitoring with MVR

It is important to establish an MVR threshold value and an MVR target value. This can be determined analytically by preparing powder blends comprising used powder and varying proportions of virgin powder and determining the respective MVR values of these blends. The proportion of virgin powder (refresh rate) shall lie within the range recommended by the material supplier and be varied above and below in increments of no more than 5 %. Build tests are then conducted using these powders and relevant part characteristics such as mechanical properties, surface quality, dimensional tolerances and detail resolution are determined. Based on the MVR values of these parts, an MVR threshold limit can be assigned to each application and each material, since powders with MVR values which produce parts whose relevant characteristics fall below a specified level are not suitable for the target application. In practice, however, this approach is extremely time-consuming.

It is also possible to measure in-line and establish a threshold value over time. This is done by measuring at regular intervals the MVR value of a powder blend with a constant refresh rate comprising used and virgin powder. Due to variations in the quality of the used powder, e.g. due to different build cycle times, it is possible to collect different MVR settings over time and correlate them directly with the production results. This requires the in-line production of test parts for the purpose of determining key component characteristics. If the actual values are close to the specification limit or even fall out of specification, the corresponding MVR values can be excluded in future. Future powder blends which have the same or a lower MVR value shall then be blended with more virgin powder to obtain a sufficiently high MVR value. In this way, an MVR threshold can be determined over time for each application.

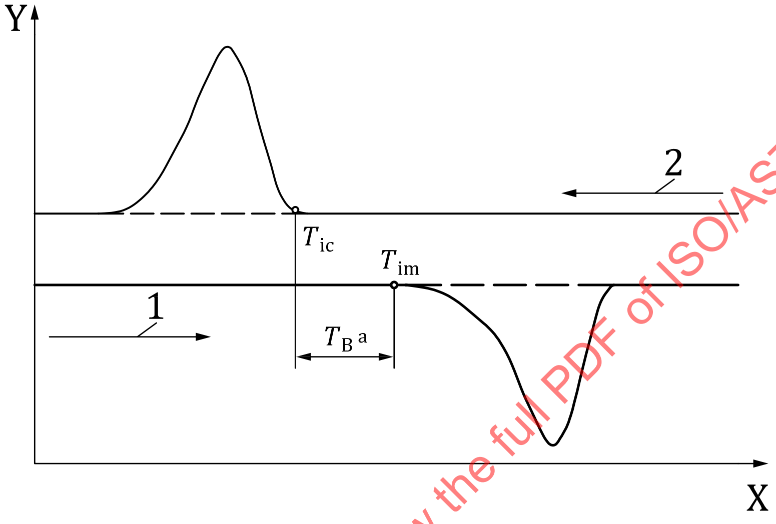
As applications change, thresholds for a specific application can also be found. In this case it is advisable to adjust the quality of the powder to suit the component characteristics required.

8.3 Melting temperature and recrystallisation temperature

In addition to the above mentioned parameters, thermal material characteristics play a vital role in PBF-LB/P. The wrong processing temperature, for instance, can lead to process errors and/or process interruptions. According to the model of quasi-isothermal PBF-LB/P, the semi-crystalline thermoplastic used, in this case PA11 and PA12, is in a two-phase mix zone throughout the entire build cycle. In other words, both the melt and the powder are present at the same time. During processing the PA11 or PA12 powder is preheated to a temperature between the initial crystallisation temperature, T_{ic}, and the initial melt temperature, T_{im}, so that the laser supplies only the energy required to melt the powder. The thermal characteristics of the powder thus determine the selected processing temperature range.

If the temperature is too high, the powder will start to melt prematurely. The sintering-on or baking of the powder is a precursor to this error, which often causes cracks to form in the powder bed. As a result, when a new layer of material is subsequently applied, the powder may be unevenly distributed or exposed structures may be pushed across the powder onto the part by the force of the powder spreading device. On the other hand, if the temperature in the build chamber is too low, crystallisation is initiated. Incipient crystallisation leads to curling, i.e. warpage of a layer or of the entire part. If this effect is too pronounced, during the powder spreading phase the powder spreading device can drag the next layer or the part along with it due to the raised areas. This would also result in termination of the build process.

With PBF-LB/P, various differential calorimetry methods (dynamic differential calorimetry, dynamic scanning calorimetry, DSC) in accordance with ASTM D3418[15] or ISO 11357-1 are used for the thermal analysis of PA11 and PA12. Heating and cooling rates of 1 K/min to 40 K/min are relevant for analysis. A heating and cooling rate of 10 K/min is normally used. During PBF-LB/P, DSC analysis is used to estimate the processing temperature range, T_B, (see Figure 1). The processing temperature for PBF-LB/P shall be verified respectively on each AM machine, since measurements performed under ideal conditions are transferable only to a limited extent.



Key

X	temperature
Y	heating flow
1	heating
2	cooling
T_{ic}	initial crystallisation temperature
T_{im}	initial melt temperature
a	Processing temperature.

Figure 1 — Example of a DSC curve of a semi-crystalline thermoplastic, e.g. PA11 or PA12

Annex A
(informative)

Hausner ratio, H_R

A.1 General

The Hausner ratio is a dimensionless value which can be used to estimate the flow characteristics of powders. It is calculated from the quotient of tapped density, ρ_0, and bulk density, ρ_∞, or bulk volume, V_∞, and tapped volume, V_0, and thus indicates the extent to which a powder can be compressed by tapping or shaking compared with the untapped, freely settled powder, as given by Formula (A.1):

$$H_R = \frac{\rho_0}{\rho_\infty} = \frac{V_\infty}{V_0} \geq 1 \qquad \text{(A.1)}$$

A.2 Determination of the Hausner ratio

Measurement of the Hausner ratio enables a statement to be made about the spreadability characteristics of a powder. A great advantage of the Hausner ratio is that it can be measured without expensive apparatus or special equipment. A plastic measuring cylinder with a volume of 100 mL to 500 mL and a suitable powder funnel are sufficient.

The following method of determining the Hausner ratio is recommended, using a graduated 250-mL measuring cylinder marked in increments of 10 mL or more accurate:

a) Pour 200 g to 300 g of the powder under investigation (test powder) into a sealable plastic bag. Knead the bag by hand to disperse the powder and break down any agglomerate.

b) Pour the test powder from a height of approximately 50 mm through the funnel into the measuring cylinder up to the 250-mL mark, avoiding any mechanical compaction (bulk volume V_∞ = 250 mL).

c) Tap the measuring cylinder by hand on a hard surface to compress the powder. Tapping requirements:

   - — tap consistently with minimal force;
   - — frequency: ~0,5 Hz;
   - — duration: 2 min.

d) Determine the provisional tapped volume V_0 to an accuracy of ~5 mL.

e) Continue tapping for a further minute and determine the tapped volume V_0 again.

f) Repeat this process until little further volume change is observed (±5 mL).

g) Calculate the Hausner ratio (single determination) using the following Formula (A.2):

$$H_R = V_\infty / V_0 \qquad \text{(A.2)}$$

where V_∞ = 250 mL, the formula is simplified as follows: H_R = 250 mL/V_0.

IMPORTANT — Points a) to g) should be performed three times using a fresh powder sample each time (triple determination). The final H_R value is derived from the mean value of the three determinations.

NOTE Compared with ISO 61 and ISO 787-11, the simplified method described here for determining the tapped volume is sufficient for the purposes of this document.

A.3 Round robin test for Hausner ratio

Two round robin tests were performed in 2013 and 2016.

Eleven laboratories took part in the first round robin test (2013). Various unfilled PA12 PBF-LB/P powders were analysed. The participating laboratories had to conduct a threefold determination for each powder. The samples received no special conditioning treatment before being analysed. Data were evaluated using robust statistical procedures based on Hampel's estimator, that is more robusts for single spike compared to other statistical analysis. Essentially, a statistical mean value (Hampel's estimator) and the repeatability standard deviation within one laboratory and reproducibility standard deviation from laboratory to laboratory for H_R were determined for each powder.

The second round robin test (2016) involved 12 laboratories and 20 datasets and was extended to filled PBF-LB/P powders and PA11 powders. This produced a sufficiently large amount of measurement data. The second round robin test was also evaluated using robust statistical procedures.

The first (2013) round robin test shows that very reproducible results can be obtained even in different laboratories and thus that free-flowing powders (= less compressible by tapping) can be clearly distinguished from poorly flowing powders. For polyamide 12, a Hausner ratio of maximum 1,25 is indicated as the limit for reliable powder processing. It is also possible to distinguish ageing differences in the same type of powder. For example, the Hausner ratio also provides initial information about the age and/or suitable blending ratio of used and virgin powder.

To supplement the 2013 round robin test, the powder test was extended to include PBF-LB/P powder mixed with fillers and polyamide 11 (PA11). Table A1 also contains the results for the standard deviation of repeatability, s_r, and reproducibility, s_R.

The standard deviations of repeatability, s_r, in Table A1 are below 1 % for the powders under investigation. The method can be performed in a laboratory with a high degree of precision. The round robin test produced values between ~3 % and 4 % for interlaboratory reproducibility of measurement data (reproducibility standard deviation s_R). The resulting s_R/s_r ratio in the 3 to 6 range shows that the Hausner ratio is suitable for the qualification of filled and unfilled PBF-LB/P powders.

Table A.1 — Hausner ratios (mean H_R values) and associated standard deviations for repeatability and reproducibility of different PBF-LB/P materials obtained in round robin tests

Material	Mean H_R	Repeatability s_r	Reproducibility s_R
Second round robin test (2016) with 250-mL measuring cylinder			
PA12 GB virgin powder	1,105	0,007 (0,64 %)	0,021 (1,86 %)
PA11virgin powder	1,205	0,007 (0,61 %)	0,048 (4,00 %)
PA12 MF virgin powder	1,231	0,009 (0,70 %)	0,060 (4,87 %)
First round robin test (2013) with 100-mL measuring cylinder			
PA12 virgin powder Type A	1,172	0,009 (0,73 %)	0,039 (3,28 %)
PA12 virgin powder Type B	1,137	0,007 (0,60 %)	0,035 (3,44 %)
PA12 used powder Type B	1,216	0,008 (0,67 %)	0,035 (2,91 %)

As a basic rule it is possible to assume, that the lower the mean H_R value, the better the powder spreadability is. The highest and lowest mean H_R value for PBF-LB/P powders with added fillers were determined in the second round robin test. This result illustrates the impact of fillers on the spreadability of the powder.

PA12 MF , a mineral fibre-filled PA12, has the highest H_R value = 1,231. PA12 GB has a mean H_R value of 1,105, which is the lowest. This can be explained by the isotropy of the additives and their aspect ratio.

The anisotropic fibres in PA12 MF prevent good compression of the powder. In contrast, the spherical glass beads in PA12 GB facilitate powder compression by mechanical tapping. It is also worth noting that at 1,205, the mean H_R value of virgin PA11 is high compared with that of virgin PA12 (Type A and B). This is due to the lower sphericity of the PA11 particles resulting from the milling process used to manufacture this powder.

Annex B
(informative)

Determination of the melt volume-flow rate (MVR)

B.1 General

When determining the MVR of semi-crystalline PBF-LB/P polymers for quality assurance purposes, a batch-wise approach is essential. To achieve this, the powder is homogenised in larger quantities and added to the PBF-LB/P process, with each batch undergoing quality control.

Determination of the MVR using Method B as specified in ISO 1133-1 is described below. For details of how to select the temperature and load, see ISO 1133-1:2011, Annex A and Annex B.

B.2 MVR measuring instrument

ISO 1133-1:2011, Clause 5 specifies an extrusion plastometer which shall be fitted with a sensor for automatically measuring displacement over time of the piston stroke for Method B. When measuring thermoplastics with a very high MVR value, the extrusion tool should be sealed in accordance with ISO 1133-1:2011, 5.2.1.5.

B.3 Conditioning the specimen

According to ISO 1133-1:2011, 6.2, the specimen shall be conditioned in accordance with the material standard. It is advisable to dry the polymer powder before performing the measurement due to its large surface area and the influence of moisture on its internal structure, which in some cases can be significant. A specimen conditioner is used for this purpose. The specimen is heated under vacuum and then purged with nitrogen. This ensures that the extrusion plastometer is charged with dry polymer powder. This conditioning process has a positive effect on the reproducibility of measurement results.

B.4 Indoor climate

Neither ISO 1133-1 nor ISO 1133-2 state that measurements shall be performed under standard atmospheric conditions. Nevertheless, the indoor climate should be kept as constant as possible to ensure the greatest degree of repeatability of the measurement results.

B.5 Precision

Due to the wide variety of different materials, ISO 1133-1:2011, Clause 11 does not provide any precision data for the method obtained in round robin tests. However, the standard assumes a variation coefficient of around ±10 % between laboratories and ±5 % within the same laboratory. To achieve a high degree of repeatability, it is important to establish the limiting conditions which influence the measured values, so that they can be accurately specified in a subsequent stage. The following procedure (see B.6) was designed to achieve a high degree of repeatability.

B.6 Procedure for determining the MVR

Figure B.1 shows a flow diagram for measuring the MVR of PA12. According to it, measurement of the MVR typically involves the following steps:

a) Weigh the powder specimens.

b) Fill the powder conditioning vessel and connect to the vacuum nozzle.

c) Apply the vacuum and put the specimen inside the heating chamber of the conditioner.

d) Wait until the final vacuum is less than 50 Pa (0,5 mbar)1).

e) Switch over to nitrogen on completion of the dwell time under vacuum.

f) Purge the specimen for 5 min.

g) 2 min before the end of the purging time:

- Remove the piston from the purging unit and insert the funnel;
- Close the extrusion nozzle of the MVR instrument.

h) At the end of the purging time:

- Remove the vacuum nozzle and fill the MVR instrument immediately;
- Press any residual powder remaining in the funnel into the instrument using a compaction rod;
- Filling shall take no longer than 60 s.

i) Insert the piston and connect the displacement sensor to it.

j) Apply the weight and start the preheating time.

k) Open the seal of the extrusion nozzle to release the polymer. This ensures that the extrusion nozzle is completely filled with polymer.

l) Close the extrusion nozzle just before reaching the lower reference mark. Adjust the process, so that this is completed within 180 s after opening the seal.

m) Open the nozzle seal on completion of the preheating time. Initiate the automatic measuring process in the MVR instrument.

B.7 Practical experience

The MVR measurement is sensitive to disruptive factors. For this reason, ISO 1133-1 and ISO 1133-2 specify large tolerance fields for measurement uncertainty. Practical tests show that a clearly specified procedure based on the one described in B.6 that additionally takes into account the respective laboratory test conditions can significantly improve reproducibility.

Provided that the same specimen conditioning and test instruments are used under identical conditions and the same procedural requirement at different laboratories, the measurement method has been shown to provide a good qualitative statement. However, it is not possible to specify an absolute value for the perfect powder blend since offset values between different laboratories can be significant.

Further studies have shown that, despite drying the specimen, the influence of absolute room humidity cannot be ruled out. The MVR value of several polymers fundamentally increases as the room humidity rises. To minimise this effect, it is advisable to perform the measurement under Class 1 standard atmosphere as defined in ISO 291. This significantly improves reproducibility.

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1) 1 bar =0,1 MPa =10^5 Pa; 1 MPa = 1 N/mm^2.

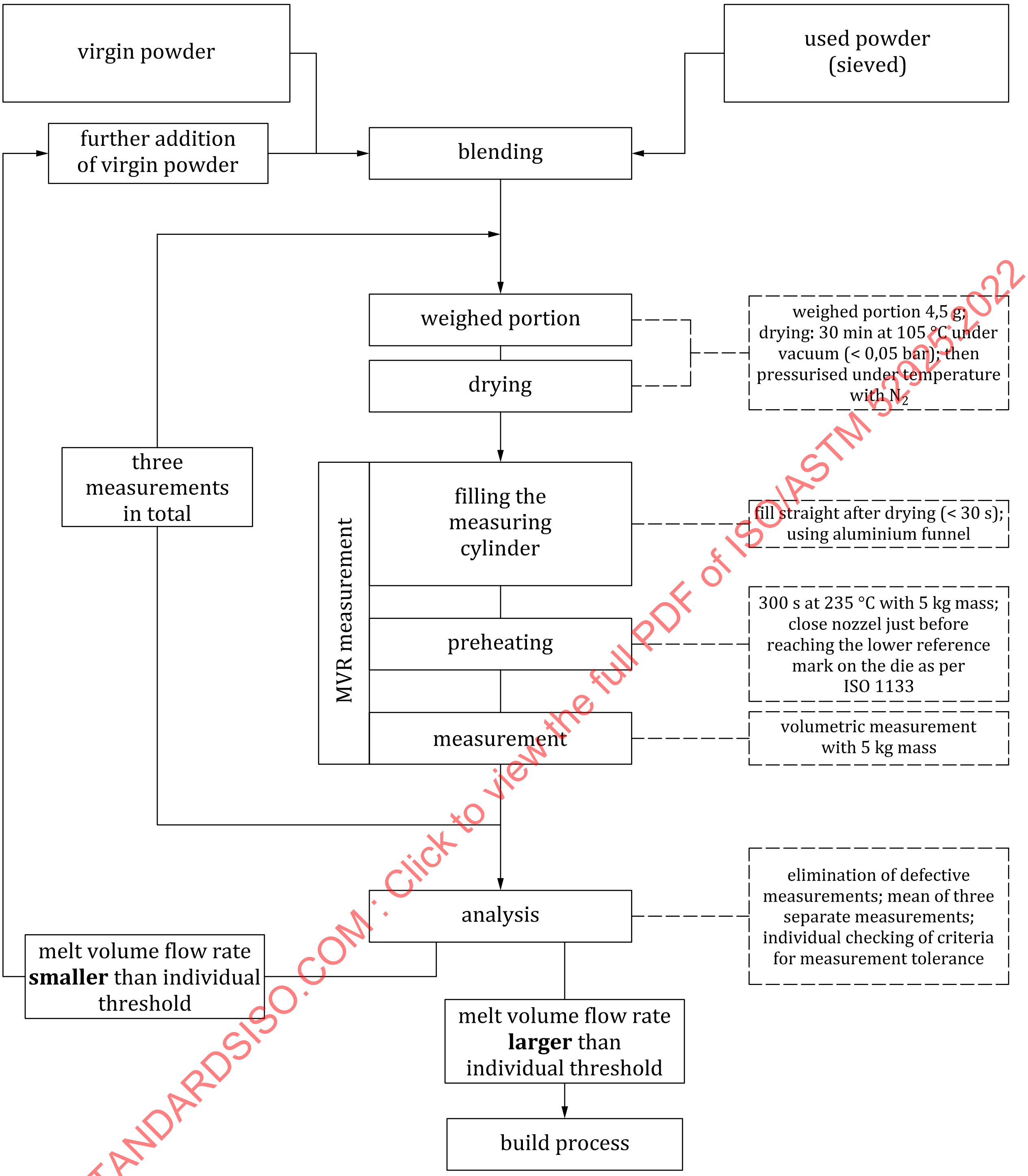

Figure B.1 — MVR measurement cycle for PA12

Annex C
(informative)

Round robin MVR test

As part of the preparation of this document, a test was conducted to study the reproducibility of MVR measurements on PA12 powders. Homogenous powder blends comprising used and virgin powder were produced in which the proportion of used powder was varied in 5 % increments from 30 % to 60 %. The samples were then divided and sent to different laboratories for analysis. All laboratories had prior experience of determining the MVR of PA12 powders and were to use their own measurement methods. The methods used by the five laboratories to prepare the samples and the process parameters used are shown in Table C.1.

Figure C.1 shows the results from four laboratories, including the respective measurement conditions. It is clearly evident that despite the different sample preparation methods, each laboratory was able to differentiate reliably between individual specimens. The curve characteristics for all measurements are also very similar and are in line with expectations that the flowability of the melt decreases almost linearly as the proportion of used powder increases. Figure C.2 shows the results from the fifth laboratory. Significantly higher MVR values were obtained here due to the different measurement conditions. Apart from that, here too the results match those from the other laboratories in qualitative terms.

Even if similar or identical measurement methods are used, it is not possible to transfer quantitative results between individual laboratories. For this reason and because the sample size (five) was insufficient for statistical analysis, a classic round robin evaluation of the results is neither sensible nor feasible.

Table C.1 — Setup used by the laboratories participating in the round robin test to dry the PA12 powder under investigation

Laboratory/participant	1	2	3	4	5
Pre-drying time	30 min	15 min/2 min	4 h	30 min	4 weeks
Pre-drying temperature in °C	105	105/150	85	105	70
Pre-drying atmosphere	vacuum	air	vacuum	air	vacuum
Measurement mass in kg	5	5	5	5	21,6
Measurement temperature in °C	235	235	235	235	210