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Additive ~~Manufacturing~~ **manufacturing** of metals — Feedstock materials — Correlation of rotating drum measurement with powder spreadability in PBF-LB machines

Fabrication additive de métaux — Matières premières — Corrélation de la mesure du tambour rotatif avec la capacité d'étalement de la poudre dans les machines PBF-LB

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Contents

Foreword	i
Introduction	v
1 Scope	1
2 Normative references	2
3 Terms and definitions	2
4 Designation	2
5 Methodology	3
5.1 Powder selection.....	3
5.2 Layer homogeneity evaluation.....	3
5.3 Rotating drum.....	4
6 Results and discussion	5
6.1 Spreadability.....	5
6.2 Rotating drum analysis.....	7
6.2.1 Experimental protocol.....	7
6.2.2 Experimental results.....	7
6.3 Discussion.....	9
7 Conclusions	11
8 Additional data	11
9 Perspectives	12
Bibliography	13
Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	2
3 Terms and definitions	2

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4	Designation.....	2
5	Methodology.....	3
5.1	Powder selection.....	3
5.2	Layer homogeneity evaluation.....	3
5.3	Rotating drum.....	4
6	Results and discussion.....	5
6.1	Spreadability.....	5
6.2	Rotating drum analysis.....	7
6.2.1	Experimental protocol.....	7
6.2.2	Experimental results.....	7
6.3	Discussion.....	9
7	Conclusions.....	11
8	Additional data.....	11
9	Perspectives.....	12
	Bibliography.....	13

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Foreword

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~~The committee responsible for this~~ This document ~~is~~ 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).

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Introduction

Granular materials and fine powders are widely used in industrial applications. To support control and optimize processing methods, these materials have to be precisely characterized. Characterization methods are related either to the properties of the grains (granulometry, morphology, chemical composition, etc.) or to the behaviour of the bulk powder (flowability, density, blend stability, electrostatic properties, etc.). The complex behaviours of granular and powder materials have motivated the development of numerous techniques to obtain reproducible and interpretable results. Many industries are concerned in different fields: additive manufacturing, food processing, pharmaceuticals, bulk material handling. This document is focused on Additive Manufacturing (AM).

Metallic powders are widely used in AM processes involving powder bed fusion (PBF-LB/M PBF-EB/M etc.) or binder jetting. During such operations, successive thin layers of powder are created with a blade or with a rotating cylinder. Each layer is then fused (most commonly melted) by an energy beam or joined by an adhesive binder to build the parts. The layer thickness defines the vertical resolution of the process; a thin layer leads to a better resolution. In order to obtain a thin layer, the powder is as fine as possible. However, if it is assumed that among the cohesive forces, the Van der Waal forces are predominant, it can be stated that as the grain size decreases, cohesiveness typically increases^[25]. This increase in cohesiveness could have an impact on the spreadability of a powder.

The quality of the parts built with AM is thus directly influenced by powder flow properties.

According to ISO/ASTM-52900, spreadability is the ability of a feedstock material to be spread out in layers that fulfil the requirements for the AM process; this includes the ability to form a strictly flat powder-atmosphere interface without waves and irregularities.

Visual observation of layer homogeneity is usually the only way for operators to assess the spreadability of powders during the spreading of new layers. However, linking the powder characteristics to its spreadability during the layer deposition beforehand can provide a more cost-effective way to classify and select the optimal powder and layer deposition speed combinations.

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Additive Manufacturing of metals — Feedstock materials — Correlation of rotating drum measurement with powder spreadability in PBF-LB machines

1 Scope

This document provides an example of the relation between the characterization of certain macroscopic properties of metallic powders and their spreadability in an PBF-LB/M AM machines.

This relation is based on a new technique combining measurements inside an PBF-LB/M machine and image processing developed to quantify the homogeneity of the powder bed layers during spreading.

In this document, the flowability of five metal powders are investigated with an automated rotating drum method, whose dynamic cohesive index measurement is shown to establish a correlation with the spreadability of the powder during the layer deposition operation. Furthermore, the particule size distribution (PSD) and morphology of each powder is characterized before testing by static image analysis method (according to ISO 13322-1:2014).

The general principle of the method is described in Figure 1.

Key

- 1 AlSi₁₂Mg
- 2 NiCr₂₂Mo₅Nb (Inconel fine)
- a Good
- b Bad
- c Rotating drum
- d PBF-LM printer
- e Regular layer
- f Irregular layer

Figure 1 — General principle of comparing rotating drum measurements with powder spreading in a PBF-LB-AM machine

2 Normative references

~~There are no normative references in this document.~~

~~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~~

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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 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/>

3.1 cohesiveness

physical powder behaviour relating to the degree to which the attractive forces between particles exceed the average particle mass

Note 1 to entry: cohesive powders is qualified as systems where the attractive force between particles exceed the average particle mass

3.2 powder flowability ability of a solid bulk material to flow

Note 1 to entry: powder flowability is a function of multiple factors, and particularly powder size and distribution, [See also ISO/ASTM-52907](#).

4 Designation

In this document, five powders described in [Table 1](#) are used:

Table 1 — Designation of powders

Common designation	European specification	Denomination used in this document
Scalmalloy®	AlMgSc	AlMgSc_Std
Inconel®	NiCr ₂₂ Mo ₉ Nb	NiCr ₂₂ Mo ₉ Nb_Std
AlSi ₇ Mg	AlSi ₇ Mg	AlSi ₇ Mg_Std
Titanium Fine	Ti ₆ Al ₄ V	Ti ₆ Al ₄ V_Fine
Inconel® Fine	NiCr ₂₂ Mo ₉ Nb	NiCr ₂₂ Mo ₉ Nb_Fine

5 Methodology

5.1 General principle

[The general principle for comparing rotating drum measurements with powder spreading in a PBF-LB AM machine is described in Figure 1.](#)

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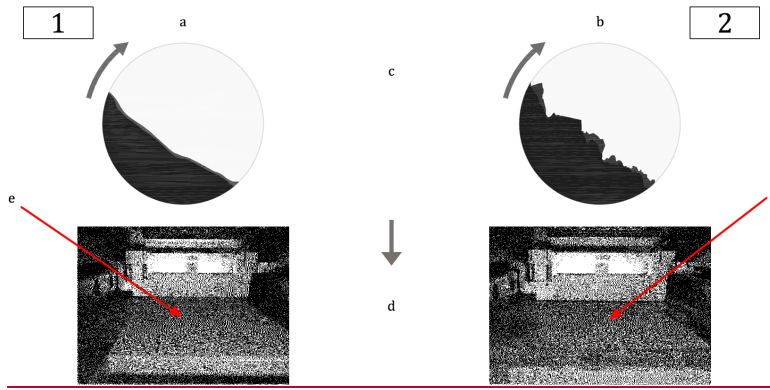
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- Key**
- 1 [AlSi7Mg](#)
 - 2 [NiCr₂₂Mo₉Nb \(inconel fine\)](#)
 - a [Good](#)
 - b [Bad](#)
 - c [Rotating drum](#)
 - d [PBF-LM printer](#)
 - e [Regular layer](#)
 - f [Irregular layer](#)

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Figure 1 — General principle of comparing rotating drum measurements with powder spreading in a PBF-LB AM machine

5.4.5.2 Powder selection

The recoating performance of the powders inside a PBF-LB AM machine is evaluated experimentally with *in situ* observation of layer homogeneity. Five metallic powders are selected for this study: two Nickel alloys (NiCr₂₂Mo₉Nb_Std and NiCr₂₂Mo₉Nb_Fine), two Aluminium alloys (AlSi₇Mg_Std and AlMgSc_Std) and one Titanium alloy (Ti₆Al₄V_Fine). Particle size distribution (PSD) is summarized in Table 2 and shape and morphology in Table 3.

Table 2 — Summary of the PSD (D10 and D90) of the five powders (volume)

Powder	D10 μm	D90 μm
NiCr ₂₂ Mo ₉ Nb_Fine	6	27
NiCr ₂₂ Mo ₉ Nb_Std	17	45
AlSi ₇ Mg_Std	27	69
AlMgSc_Std	26	66
Ti ₆ Al ₄ V_Fine	7	28

Table 3 — Shape and morphology comparison

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Aspect ratio comparison				
Aspect ratio (number)	Mean (µm)	P10 (µm)	P50 (µm)	P90 (µm)
AlMgSc_Std	79,7	62,5	81,6	93,8
AlSi7Mg	76,6	58,4	78,7	91,8
NiCr22Mo9Nb_Std	81,9	63,5	85,3	94,6
NiCr22Mo9Nb_Fine	81,8	63,5	85,8	93,3
Ti6Al4V_Fine	79,7	60,9	82,9	92,8
Bluntness comparison				
Bluntness (number)	Mean (µm)	P10 (µm)	P50 (µm)	P90 (µm)
AlMgSc_Std	74,6	54,0	74,6	95,1
AlSi7Mg	75,5	57,1	75,5	93,8
NiCr22Mo9Nb_Std	84,3	67,9	86,7	97,2
NiCr22Mo9Nb_Fine	88,0	76,5	89,9	97,1
Ti6Al4V_Fine	85,0	70,2	87,4	96,5

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Successive powder layers are produced in the PBF-LB AM machine with no laser melting. Between each layer deposition, a picture of the powder layer is taken by a camera placed inside the AM machine. The pictures are then processed numerically to evaluate the layer homogeneity. Three powder spreading speeds are investigated: 30, 80 and 160 mm/s to highlight their influence on the layer quality.

5.25.3 Layer homogeneity evaluation

The powder layer surface homogeneity is experimentally evaluated using a camera placed orthogonal to the powder bed. After each powder spreading operation, a picture is taken. For this experiment, the focus is made on metallic coater and 30 µm layer thickness only. For the same recoater speed, 15 layers are created and therefore, 15 pictures are taken as well. This methodology provides a quantitative and operator independent way to quantify the layer topography homogeneity.

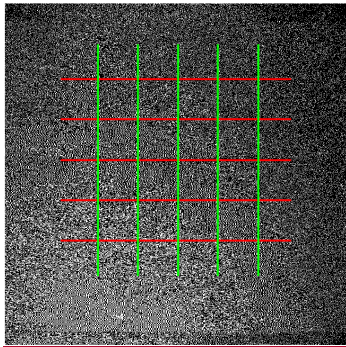
The gathered pictures are then processed numerically to obtain "Interface Fluctuation", a measure of the inhomogeneity of the produced layers. The image processing analysis principle is as follow:

- a) each picture is analysed separately. The picture size is 1 200 ~~pixels~~ × 1 200 ~~pixel~~pixels;
- b) horizontal and vertical pixel intensity profiles are extracted at discrete positions of the picture (see Figure 2- a));
- c) an average "smooth" profile is computed for each position [see Figure 2- b)];
- d) interface fluctuation is then computed based on the deviation around the averaged profile, and then averaged over all positions;
- e) the process is repeated for all the images, and the interface fluctuation is average over the whole set of pictures.

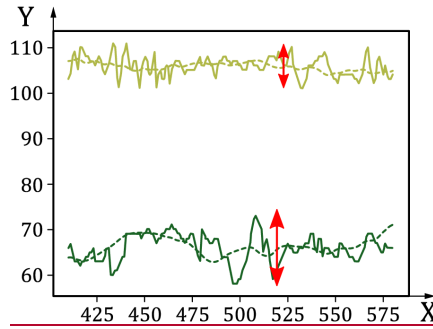
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a) Horizontal and vertical lines from which pixel intensity profiles are extracted



b) Pixel intensity profile (plain) and average profile (dashed) used to compute the interface fluctuation

Key

- X position along the line
- Y pixel intensity (before normalisation)

52952_e
 d1fig2_k AlSi₇Mg (top)
 ey1.EPS

52952_e
 d1fig2_k NiCr₂₂Mo₉Nb_Fine (bottom)
 ey2.EPS

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Figure 2 — In situ layer quality assessment

5.35.4 Rotating drum

Powder flowability is evaluated with a rotating drum method which allows an automated measurement.

A horizontal cylinder with transparent sidewalls called drum is filled with the sample of powder. The filling ratio of the drum can influence the flow of the powder and thus are kept constant to allow relevant comparison of the results.

The volume of the drum used in this study is 100 mL and the filling ratio is fixed as 50 %. Therefore, a 50 mL powder sample is used for the measurements.

The drum rotates around its axis at an angular velocity ranging from 2 ~~rpm/min~~ to 60 ~~rpm/min~~. A CCD (charge-coupled device) camera takes snapshots at a framerate of 1 image per second for each angular velocity.

The air/powder interface is detected on each snapshot with an edge detection algorithm.

Afterwards, the average interface position and the fluctuations around this average position are computed. The number of snapshots taken influences the statistical relevance of the averaged interface.