INTERNATIONAL STANDARD

ISO 14232-1

First edition 2017-03

Thermal spraying — Powders —

Part 1:

Characterization and technical supply conditions

Projection thermique — Poudres —

Partie 1: Caractérisitation et conditions techniques de livraison





COPYRIGHT PROTECTED DOCUMENT

© ISO 2017, Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office Ch. de Blandonnet 8 • CP 401 CH-1214 Vernier, Geneva, Switzerland Tel. +41 22 749 01 11 Fax +41 22 749 09 47 copyright@iso.org www.iso.org

Contents Page Foreword iv Introduction v 1 Scope 1 2 Normative references 1 3 Terms and definitions 1 4 Properties and property determination of powders for thermal spraying ______1 Sampling and sample splitting ______1 4.1 4.2 Chemical composition 2 4.3 Manufacturing process — Particle shape ______3 4.4 4.5 Apparent density 3 4.6 Flowability 3 4.7 Microstructure 4 4.8 4.9 Summary......4 5 6 Powder identification and designation 4 7 Conditions of supply 4 Certificate......5 8 Annex A (informative) Powder shape and morphologies......6 Bibliography 9

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on 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 the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*.

This first edition of ISO 14232-1, together with ISO/TR 14232-2, cancels and replaces ISO 14232:2000.

A list of all parts in the ISO 14232 series can be found on the ISO website.

Introduction

For thermal spray processes that use material in a powder form, the powder can be considered as one of the main elements requiring control to produce an acceptable coating. The chemical and physical properties of powder play a fundamental role in the creation of desired coating properties. The size, shape and morphology of the powder particles determine the melting behaviour and therefore the processing of powders in general.

To keep the coating properties and the spray process as consistent as possible, it is very important to maintain all the characteristics of powder particles within limited tolerances.

An exception is granted to details on the properties of sprayed coatings. Such properties, which result from spraying conditions not covered by this document, e.g. gas composition, deposition efficiency, material flow rate, stand-off distance, etc., can differ greatly from the properties of the original powder.

The ISO 14232 series consists of two parts. This document examines the characterization of spray powders. ISO/TR 14232-2 is a technical report that examines how technical literature describes the application of powders.

Thermal spraying — Powders —

Part 1:

Characterization and technical supply conditions

1 Scope

This document specifies measuring methods for the characterization of powders and their technical supply conditions. It is applicable to powders that are used in thermal spraying on the basis of their physical and chemical properties.

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 3923-2, Metallic powders — Determination of apparent density — Part 2: Scott volumeter method

ISO 3954, Powders for powder metallurgical purposes — Sampling

ISO 4490, Metallic powders — Determination of flow rate by means of a calibrated funnel (Hall flowmeter)

ISO 10474, Steel and steel products — Inspection documents

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 14917 and the following 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

3.1

particle size distribution

PSD

distribution of particle sizes in percentage within standard ranges

3.2

particle size range

range of particle sizes between an upper and lower limit

4 Properties and property determination of powders for thermal spraying

4.1 Sampling and sample splitting

Sampling and sample splitting is to be done from a homogeneous mixture uniform in particle size. Directions for adequate methods and equipment shall be as included in ISO 3954.

4.2 Chemical composition

The chemical composition shall be defined by any suitable testing procedure, e.g. ICP-OES (inductively coupled plasma optical emission spectrometry), wet chemical processes, atomic absorption spectrometry, flame emission spectroscopy, X-ray fluorescent analysis and energy dispersive X-ray spectroscopy (EDX).

4.3 Particle size range — Particle size distribution

Powders for thermal spraying always show a distribution of different particle sizes. This particle size distribution (PSD) has an immense influence on the melting and the feedability of the powder and thus, among other things, essential properties of the coating are assigned. The measuring of the PSD shall be made by sieve analysis, air-jet sieving, etc. or by optical measuring methods like laser scattering because of its high accuracy and reproducibility.

Particle size ranges are documented in specifications by upper and lower size limits like " $(125 \, \mu m/5 \, \mu m)$ ".

Usually, the results of different measuring procedures and also measuring devices do not coincide, even when identical powders are used. Therefore, when comparing particle size distributions or when changing the particle size measuring procedure, a correlation of the measurement results is necessary. Therefore, it is essential to always indicate the measuring procedure together with the measurement results. Coarse powders (125 $\mu m/38~\mu m)$ can be characterized by sieving methods, while for fine powders (45 $\mu m/5~\mu m$), laser scattering is more suitable.

Sizing methods used for spray powder characterization are restricted in their accuracy dependent on the absolute size of powders. The following limitations for oversized and undersized particles are common in the industry:

— Upper size limit (range from 125 μm to 15 μm):

Sieving method (used for size range 125 μ m to 38 μ m): max. 10 % coarser than the indicated upper limit, see footnote ^a in Table 1;

Sieving method "air-jet" (used for size range 45 μ m to 5 μ m): max. 5 % coarser than the upper size limit, see footnote ^a in Table 1;

Laser scattering: (used for size range 63 μm to 15 μm): max. 25 % coarser than the upper size limit, see footnote b in Table 1;

— Lower size limit (range 90 μ m to 5 μ m):

Sieving method (used for size range 90 μm to 38 μm): max. 10 % undersized particles, see footnote ^a in Table 1;

Sieving method "air-jet" (used for size range 38 μ m to 5 μ m): max. 10 % undersized particles, see footnote ^c in Table 1;

Laser scattering (used for size range 53 μm to 5 μm): max. 5 % undersized particles, see footnote ^c in Table 1.

EXAMPLE Size designation 63/38 μ m; Means: If measured by sieving: max. 10 % coarser than 63 μ m and max. 10 % finer than 38 μ m; 0 % coarser than 90 μ m and max. 1 % finer than 30 μ m. "Air-jet" is usually not used for this size range. If measured by laser scattering: max. 25 % coarser than 63 μ m and max. 5 % coarser than 90 μ m; max. 1 % finer than 30 μ m. For further clarification, see the given examples in Table 1.

Measuring method	Upper size limit		Remarks	Lower size limit	
	(range 125	μm to 15 μm)		(range 90 μm to 5 μm)	
	Particle size range		Coarser at next but one higher standard screen	Particle size range	Undersized particles ^c
Sieving method	125 μm to 38 μm	max. 10 % ^a	coarsest particles 0 %	90 μm to 38 μm	max. 10 %
Sieving method "air-jet"	45 μm to 5 μm	max. 5 % ^a	coarsest particles 0 %	38 μm to 5 μm	max. 10 %
Laser scatter- ing	63 μm to 15 μm	max. 25 % ^b	max. 5 %	53 μm to 5 μm	max. 5 %

Table 1 — Particle size range

It should be noted, when evaluating powders for HVOF (high-velocity oxygen fuel), special attention should be paid to fine particles (dust) because of their effect on the feeding and melting properties of the powder. Further, these fine dust particles can clog the spray nozzle and so stop the whole coating process. This, however, often leads to increased manufacturing costs of powder. These additional requirements in the measuring and the description of the measured values can exceed the minimum requirements of this document and shall be stipulated between the powder supplier and the user in the technical delivery conditions.

4.4 Manufacturing process — Particle shape

The manufacturing process of powder shall be indicated using a term such as fusing (f), sintering (s), agglomeration (ag), gas or water atomization (gat, wat), coating (c), spheroidization (sp), blending (bld). The particle shape and its surface can be illustrated by means of scanning electron or stereo microscopy. In order to check for similarity, the images can be compared to reference samples provided by the manufacturer.

Example figures are included in Annex A.

4.5 Apparent density

Apparent powder density shall be determined as specified in ISO 3923-2, and to be expressed as g/cm³.

Alternatively, ASTM-B 329, ASTM-B 417 and ASTM-B 212 can be used to analyse the apparent density.

4.6 Flowability

The determination of the flowability of free-flowing powders shall be according to ISO 4490 and expressed as s/50 g. Fine powders with a low specific weight are often not able to flow and it is not possible to determine the flowability in these cases.

Alternatively, ASTM-B 213 can be used to analyse flowability. For blends containing organic powder materials, specific flowability measurement methods are applied. These methods shall be documented in the certificate.

The results of the measurements of apparent density and flowability are determined by several properties of the material; for example, specific weight, shape and structure of the powder particle and size distribution. Corresponding measured values and tolerances are to be stipulated between the supplier and the user if these characteristics of the powder shall be stated in the technical delivery conditions.

a Coarsest particles 0 % at next but one higher nominal size of (sieve/subsieve) opening.

b Max. 5 % coarser at next but one higher nominal size of (sieve/subsieve) opening.

 $[^]c$ Finest particles max. 1 % at next but one lower nominal size of (sieve/subsieve) opening. Nominal sizes of (sieve/subsieve) openings are: 5 μm , 10 μm , 16 μm , 20 μm , 25 μm , 32 μm , 45 μm , 53 μm , 63 μm , 75 μm , 90 μm , 106 μm , 125 μm , 150 μm , 180 μm .

4.7 Microstructure

The microstructure of a powder particle can be represented in a metallographically prepared cross-section. The preparation method is of decisive importance, and should, therefore, be agreed upon between the manufacturer and the user.

4.8 Determination and composition of phases

Determination of phases regarding type, quantity, shape, configuration, composition and size, in multiphase powders can be made by X-ray microstructure analysis, scanning electron microscope (SEM), energy dispersive X-ray analysis (EDX), metallographic or quantitative image analysis. If required, the method of determination shall be agreed upon the contracting parties.

4.9 Summary

Each powder for thermal spraying according to this document shall be characterized at least by the chemical composition, particle size distribution measuring procedure and manufacturing process of the powder.

5 Classification of powders

The powders for thermal spraying are categorized based on their chemical composition into the following:

- pure metals, e.g. Mo, Cu, Ni, Ti, etc.;
- metallic alloys, e.g. group of self-fluxing alloys, NiCr, MCrAlY ("M" stands for metals like Ni, Co or Fe), bronze, AlSi, steels, NiAl, CoCr-alloys, etc.;
- composites and blends, e.g. Ni-C, AlSi-polyester, WC-Co/Ni-SF, etc.;
- carbides, carbides with metals, carbides with metallic alloys, e.g. WC-Co, CrC-NiCr, WCCoCr, WCrCNi, etc.;
- oxides, e.g. Al₂O₃, Al₂O₃-TiO₂, Cr₂O₃, TiO₂, ZrO₂-Y₂O₃, etc.;
- organic materials, e.g. polyester, ethylene-hydroethylene-copolymer, etc.

The chemical composition of powders are given in mass (or weight) percentage. The above given abbreviations refer to the table of elements or commonly used technical literature.

6 Powder identification and designation

The uniform identification of powders for thermal spraying should serve to designate a spray powder in a brief and well-defined (unmistakable) form.

EXAMPLE Spray powder ISO 14232-1:2017 XYZ – 45/5 measured with laser scattering – sintered; Means: XYZ stands for a commonly used chemical composition, e.g. "WC-Co 88/12 or Cr_2O_3 " or a powder supplier designation.

7 Conditions of supply

Powders shall be dry and free of contamination. Special types of packing, for example, vacuum chambers with flange connection, shall be agreed upon separately with the powder manufacturer or supplier, as applicable.

Powder containers shall be labelled "Blend thoroughly before use" and "For powder processing, the materials safety datasheet shall be considered".

The materials safety datasheet (relevant to the country to which the powder is delivered to) should either be provided with each delivery or the route of accessing this information shall be clearly defined by the supplier.

8 Certificate

The manufacturer/supplier shall certify that every batch of its product meets the requirements of the specification, documented by a certificate accompanying the thermal spray powder according to ISO 10474 (alternatively EN 10204 can be used for inspection certificates).

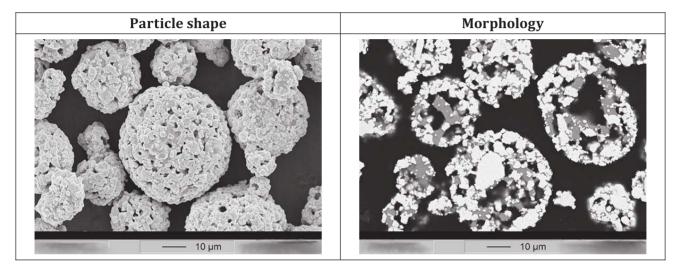
This certificate, in addition to the results of chemical and physical analysis, as far as specified and/or agreed upon between customer and manufacturer/supplier, shall contain the product designation and batch number.

The certificate shall be enclosed to the powder delivery.

Annex A (informative)

Powder shape and morphologies

Figures A.1 and A.2 show typical powder shapes and morphologies which can vary dependent on the powder manufacturing parameter.



 $Figure \ A.1 - Typical \ powder \ shapes \ and \ morphologies - Agglomerated \ and \ sintered$

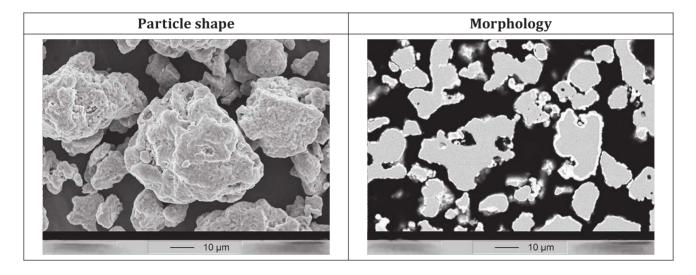


Figure A.2 — Typical powder shapes and morphologies — Dense coated

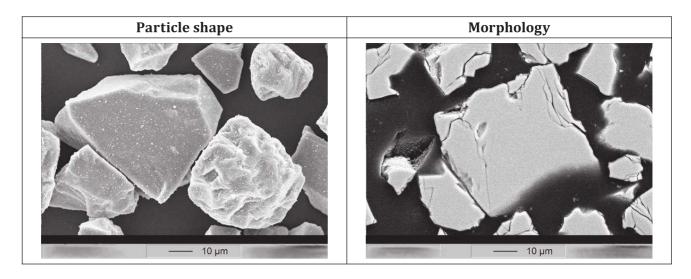


Figure A.3 — Typical powder shapes and morphologies — Fused

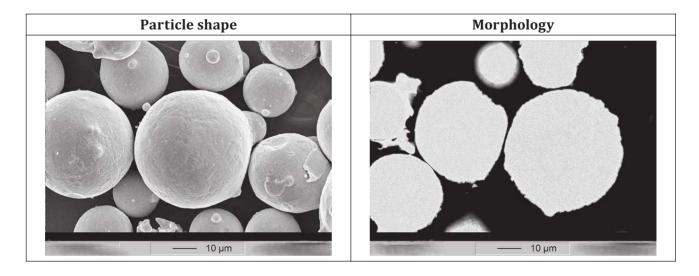


Figure A.4 — Typical powder shapes and morphologies — Gas atomized

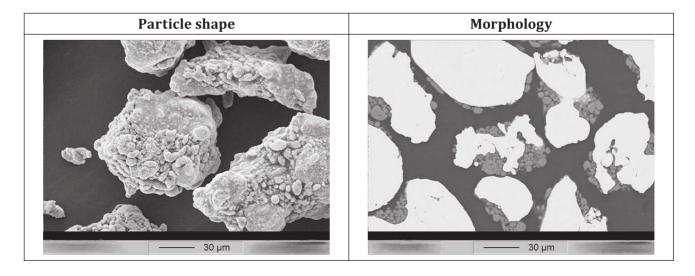


Figure A.5 — Typical powder shapes and morphologies — Porous coated

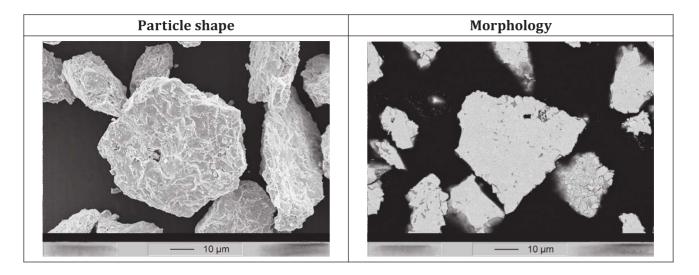


Figure A.6 — Typical powder shapes and morphologies — Sintered

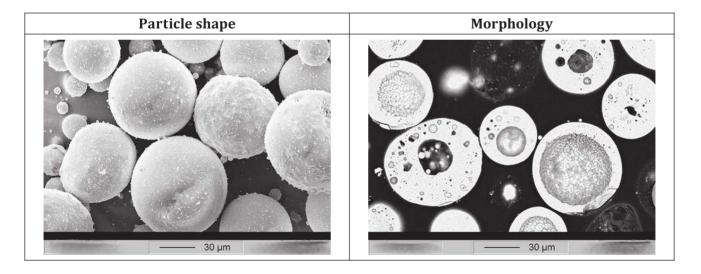


Figure A.7 — Typical powder shapes and morphologies — Spheroidized

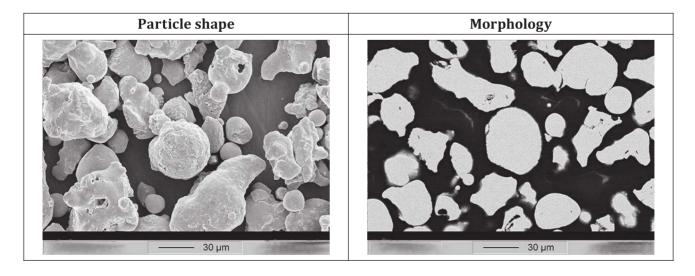


Figure A.8 — Typical powder shapes and morphologies — Water atomized

Bibliography

- [1] ISO 565, Test sieves Metal wire cloth, perforated metal plate and electroformed sheet Nominal sizes of openings
- [2] ISO 3923-1, Metallic powders Determination of apparent density Part 1: Funnel method
- [3] ISO 3953, Metallic powders Determination of tap density
- [4] ISO 4324, Surface active agents Powders and granules Measurement of the angle of repose
- [5] ISO 4497, Metallic powders Determination of particle size by dry sieving
- [6] ISO 8130-5, Coating powders Part 5: Determination of flow properties of a powder/air mixture
- [7] ISO/TR 14232-2^{1),}Thermal spraying Powders Part 2: Comparison of coating performance and spray powder chemistry
- [8] ISO 14887, Sample preparation Dispersing procedures for powders in liquids
- [9] ISO 14917, Thermal spraying Terminology, classification
- [10] ASTM-B 212, Standard test method for apparent density of free-flowing metal powders using the hall flowmeter funnel
- [11] ASTM-B 213, Standard test methods for flow rate of metal powders using the hall flowmeter funnel
- [12] ASTM-B 214, Standard test method for sieve analysis of metal powders
- [13] ASTM-B 329, Standard test method for apparent density of metal powders and compounds using the Scott Volumeter
- [14] ASTM-B 330, Standard test methods for estimating average particle size of metal powders and related compounds using air permeability
- [15] ASTM-B 417, Standard test method for apparent density of non-free-flowing metal powders using the carney funnel
- [16] ASTM-B 430, Standard test method for particle size distribution of refractory metal powders and related compounds by turbidimetry
- [17] ASTM-B 527, Standard test method for tap density of metal powders and compounds
- [18] ASTM-B 761, Standard test method for particle size distribution of metal powders and related compounds by X-ray monitoring of gravity sedimentation
- [19] ASTM-B 822, Standard test method for particle size distribution of metal powders and related compounds by light scattering
- [20] ASTM-C 721, Standard test methods for estimating average particle size of alumina and silica powders by air permeability
- [21] ASTM-C 925, Standard guide for precision electroformed wet sieve analysis of nonplastic ceramic powders
- [22] ASTM-F 1377a, Standard specification for cobalt-28 chromium-6 molybdenum powder for coating of orthopedic implants (UNS-R30075)

¹⁾ Under preparation. Stage at the time of publication: ISO/DTR 14232-2:2017.

ISO 14232-1:2017(E)

- [23] ASTM-F 1580, Standard specification for titanium and titanium-6 aluminum-4 vanadium alloy powders for coatings of surgical implants
- [24] EN 10204, Metallic products Types of inspection documents

