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Metallic powders — Test method for the determination of non-metallic inclusions in metal powders using a powder-forged specimen

Poudres métalliques — Détermination de la teneur en inclusions non métalliques dans les poudres métalliques à l'aide d'une éprouvette forgée de poudre

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 13947 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*, Subcommittee SC 2, *Sampling and testing methods for powders (including powders for hardmetals)*.

This second edition cancels and replaces the first edition (ISO 13947:2007), of which it constitutes a minor revision.

Metallic powders — Test method for the determination of non-metallic inclusions in metal powders using a powder-forged specimen

SAFETY PRECAUTIONS — This International Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a metallographic method for determining the non-metallic inclusion level in metal powders using a powder-forged specimen. The test method covers repress powder-forged test specimens in which there has been minimal lateral flow (<1 %). The core region of the powder-forged test specimen contains no porosity detectable at 100× magnification.

This test method can also be used to determine the non-metallic inclusion content of powder-forged steel parts. However, in parts where there has been a significant amount of material flow, the near-neighbour separation distance needs to be changed, or the inclusion sizes agreed between the parties need to be adjusted.

This test method is not suitable for determining the non-metallic inclusion level of parts that have been forged such that the core region contains porosity. At the magnification used for this test method, residual porosity is hard to distinguish from inclusions. Too much residual porosity makes a meaningful assessment of the inclusion population impossible.

This test method can be applied to materials that contain manganese sulfide (admixed or prealloyed), provided the near-neighbour separation distance is changed from 30 µm to 15 µm.

2 Normative references

The following referenced documents are indispensable for the application 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/TR 14321:1997, *Sintered metal materials, excluding hardmetals — Metallographic preparation and examination*

ASTM B796, *Standard test method for nonmetallic inclusion content of powders intended for powder forging (P/F) applications*

ASTM E3, *Standard guide for preparation of metallographic specimens*

ASTM E768, *Standard guide for preparing and evaluating specimens for automatic inclusion assessment of steel*

3 Principles

A section representing the core region of the test specimen (part) is cut from the test piece (part) prior to metallographic grinding and polishing (mounting the section is optional but strongly recommended).

The polished sample is examined microscopically at a magnification of 100× and a note is made of inclusions greater than a predetermined size.

The maximum Feret's diameter is used to determine inclusion size. A Feret's diameter is a caliper diameter, as illustrated in Figure 1.

The fragmented nature of some inclusions means that their size determination is somewhat complicated. The concept of near-neighbour separation shall be used in determining inclusion size. According to this concept, if an inclusion is within a certain distance of its neighbouring particles, it is considered a member of an inclusion cluster or agglomerate. Detected features within 30 µm of one another are considered part of the same inclusion. The concept is illustrated schematically in Figure 2.

The non-metallic inclusion level of the test specimen (part) is reported as the number of inclusions, per 100 mm^2, greater than or equal to the predetermined size.

4 Significance and use

4.1 The non-metallic inclusion level of ferrous powders is an indication of powder cleanliness. In pressed and sintered ferrous powder metallurgy (PM) materials, the extensive porosity present masks the effect of inclusions on mechanical properties. In contrast, the properties of a PM material processed to near pore-free density are strongly influenced by the composition, size, size distribution, and location of non-metallic inclusions.

4.2 The test for non-metallic inclusions in fully dense steel parts is useful as

— a characteristic to classify or differentiate one grade of powder or batch of parts from another, and

— a lot-to-lot quality comparison of powders intended for powder forging.

4.3 A significant amount of variation in non-metallic inclusion content will occur

— if the powder used to form the test specimen (part) does not meet the required quality standards for non-metallic inclusion content, or

— processing of the test specimen (part) has been carried out under conditions that do not permit oxide reduction or allow oxidation of the test specimen (part), or both.

4.4 The use of the near-neighbour concept offers a more conservative estimate of the inclusions: it will overestimate rather than underestimate them.

5 Apparatus

5.1 **Equipment**, for the metallographic preparation of test specimens.

5.2 **Metallographic microscope**, permitting observation and measurement up to a magnification of 100× using light with a wavelength of 544 nm (green filter), an objective lens with a magnification ranging from 8× to 12,5×, and a numerical aperture between 0,16 and 0,20.

NOTE Defining the light optics used is important because this determines the features that will be resolved, and all detected features are included in the assessment of inclusion size.

6 Test piece

6.1 For the evaluation of the non-metallic inclusion content of a powder that is intended for use in powder-forging applications, the powder shall be mixed with an appropriate amount of graphite and lubricant and compacted to a specified green density. The test method covers repress powder-forged test specimens in which there has been minimal lateral flow (<1 %) so the diameter of the green compact shall be such, relative to the diameter of the forging die, that this requirement is met.

6.2 A metallographic specimen shall be removed from the powder-forged test specimen (part), austenitized and quenched. Hardening of the specimen facilitates sample preparation by preventing smearing of soft inclusions and scratching.

6.3 The polished surface of the specimen (part) to be examined shall be not less than 350 mm^2 in area. Multiple sections are permitted in order to obtain the necessary area for measurement on small parts.

6.4 The polished surface shall be parallel to the direction of working; that is, parallel to the direction of travel of the forging tools, and shall represent the core region of the test specimen (part).

7 Procedure

7.1 Preparation of specimens

In polishing the specimens, it is highly important that a clean polish be obtained and that the inclusions not be pitted, dragged or obscured. It is therefore recommended that the procedures described in ASTM E3 and ASTM E768 be followed. Automated grinding and polishing procedures are recommended. Examine specimens in the as-polished condition, free of the effects of any prior etching (if used). ISO/TR 14321:1997 shall be followed.

7.2 Measurement of non-metallic inclusion content

7.2.1 Survey at least 350 mm^2 of the surface of the polished specimen at a magnification of 100× using light with a wavelength of 544 nm (green filter), an objective lens with a magnification ranging from 8× to 12,5× and a numerical aperture between 0,16 and 0,20.

7.2.2 Size the detected inclusions on the basis of near-neighbour separation.

Features within 30 µm (0,03 mm) of one another (within 3 mm of one another at 100× magnification) are considered to be part of the same inclusion.

7.2.3 For individual features of less than 30 µm in size, three such features within 30 µm of one another are required to constitute an inclusion aggregate, when the near-neighbour separation principle is used.

7.2.4 Add an individual feature of less than 30 µm in size to an inclusion larger than 30 µm, provided both features are within 30 µm of one another, when the near-neighbour separation principle is used. Examples are given in Figure 2.

7.2.5 Measure and record the number of inclusion particles, according to the principle of near-neighbour separation and sized using the maximum Feret's diameter, that are

— greater than or equal to 30 µm in length,

— greater than or equal to 100 µm in length, and

— greater than or equal to 150 µm in length.

In addition to the use of the near-neighbour separation concept to size the inclusions, the size of the individual particles can be measured.

7.2.6 If the inclusions are also sized individually, the inclusions are sized using the maximum Feret's diameter as in 7.2.5. Results from the use of the near-neighbour separation concept will usually be more conservative (more larger inclusions).

8 Test report

The test report should include the following information:

a) a reference to this International Standard, i.e. ISO 13947:2011;

b) the number of non-metallic inclusions per 100 mm^2 that are

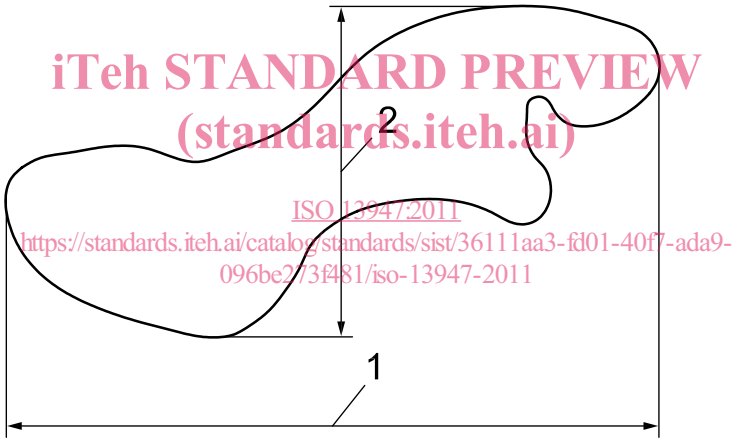
- greater than or equal to 30 µm in length,
- greater than or equal to 100 µm in length, or
- greater than or equal to 150 µm in length;

c) whether the particles were sized individually, in addition to using the near-neighbour separation principle;

d) the total area examined.

9 Precision and bias

The precision and bias that can be expected through use of this test method are currently under review by ASTM Subcommittee B09.11 on near-full-dense PM materials.

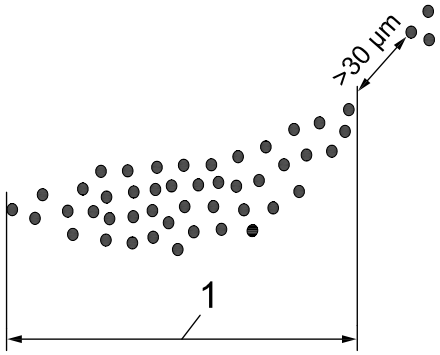


Key

1 maximum Feret's diameter

2 Feret's diameter

Figure 1 — Schematic illustration of Feret's diameter



Key

1 maximum Feret's diameter

Figure 2 — Schematic illustration of "near-neighbour" concept and maximum Feret's diameter