INTERNATIONAL STANDARD

ISO 14168

Second edition
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Metallic powders, excluding hardmetals — Method for testing copper-base infiltrating powders

Poudres métalliques, à l'exclusion des métaux-durs — Méthode d'essai des poudres infiltrantes à base cuivre

Reference number
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Foreword

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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 14168 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 14168:2003), of which it constitutes a minor revision.

Introduction

Sintered materials generally have a porous structure. There is, however, one group of metallic materials in which the porosity is greatly reduced by filling the open pores with lower-melting-point alloys, a process made possible by the capillary action of the pores. This group of materials is called infiltrated materials.

In most cases, this process is carried out as the infiltration of copper or copper-base alloys into higher-melting-point skeletons.

Metallic powders, excluding hardmetals — Method for testing copper-base infiltrating powders

1 Scope

This International Standard specifies a method for determining the performance characteristics of copper-base infiltrating powders.

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 2738, *Sintered metal materials, excluding hardmetals — Permeable sintered metal materials — Determination of density, oil content and open porosity*

ISO 3325, *Sintered metal materials, excluding hardmetals — Determination of transverse rupture strength*

ISO 3927, *Metallic powders, excluding powders for hardmetals — Determination of compressibility in uniaxial compression*

ISO 3995, *Metallic powders — Determination of green strength by transverse rupture of rectangular compacts*

ISO 4492, *Metallic powders, excluding powders for hardmetals — Determination of dimensional changes associated with compacting and sintering*

ISO 4498, *Sintered metal materials, excluding hardmetals — Determination of apparent hardness and microhardness*

3 Principle

The infiltrant is placed in contact with a skeleton, usually of iron-base composition, and both components are heated above the melting point of the infiltrant.

The infiltration efficiency and loose residue are calculated.

4 Apparatus

4.1 **Compression testing machine or hydraulic powder press**, capable of applying the required pressure.

4.2 **Compacting tools**, for producing the required specimens and infiltrant compacts.

4.3 **Furnace**, muffle type or equivalent, having a heating chamber capable of maintaining a temperature of 1 120 °C ± 8 °C; it shall have a reducing atmosphere in the hot zone while maintaining a non-oxidizing atmosphere in the cooling chamber.

4.4 **Balance**, suitable for weighing accurately to within 0,01 g.

4.5 **Micrometers**, capable of measuring to 0,005 mm.

5 Test specimens

5.1 To test an infiltrating powder, the preferred skeleton to be infiltrated is 25 mm diameter by 12,7 mm height (see ISO 3927). The skeleton may be infiltrated simultaneously with sintering, or in a separate step after sintering. The use of a one-step or two-step infiltration shall be a matter of agreement between parties, since final part properties depend on the infiltration steps.

The composition of the skeletons, including the type of material and the green density, shall also be a matter of agreement between the parties concerned. The uniformity of the skeletons may be determined by measuring the mass. The mass shall not vary from the mean by more than ±0,5 %.

5.2 The recommended infiltrant compact is a cylinder nominally 12,7 mm in diameter compacted from a copper or copper-base infiltrant mix. Normally the infiltrant mix shall also contain a dry lubricant. The composition of the infiltrant, including the type of lubricant and the mass and green density of the infiltrant compact, shall be a matter of agreement between the parties concerned. The uniformity of the infiltrant compacts shall be determined by weighing. The mass of any compact shall not vary by more than 0,5 % from the mean.

5.3 The recommended infiltrant compact, when placed on the skeleton, has a contact area of 126,5 mm^2. Since the infiltrant loading mass per unit contact area can affect the test results, this shall be a matter of agreement between the parties and shall be reported in the test report.

5.4 An alternative specimen is the transverse rupture bar described in ISO 3995, ISO 4492 and ISO 3325. The advantage of using this specimen is that the transverse rupture strength can be measured, as well as the infiltrant efficiency.

5.5 Alternatively, skeleton specimens may be made of any iron-base structural piece, of composition and sintered or green density that are acceptable to the parties concerned. Likewise, the infiltrant compact may be any available shape that is acceptable to the parties concerned, with composition, lubricant, mass and green density a matter for agreement between the parties concerned. The mass of the skeleton and infiltrant compact shall not vary by more than 0,5 % from the mean.

6 Procedure

6.1 Weigh the skeleton specimens and accurately measure the dimensions. Three specimens for each infiltrant being evaluated shall be prepared.

The density of the skeleton is determined either by calculation from the mass and dimensions or by weighing in air and water as described in ISO 2738. If the method of ISO 2738 is used to determine the density, do not use any specimen that was used for water displacement density testing for subsequent infiltration. In this case, three additional specimens shall be made for the density measurement. These three specimens shall be made of the same powder mix and according to the same procedure as the skeleton specimens to be used for the infiltration. The reported density of the skeleton specimens shall be the calculated mean value of the three specimens.

The skeletons are placed on a suitable tray or container for transporting the specimens through the infiltrating furnace. The type of tray or container shall be agreed upon by the interested parties. Care shall be taken to avoid contact with materials that would react with the skeleton or the infiltrant. The skeletons are normally positioned with one punch face surface facing up.

6.2 The weighed infiltrant compact shall be approximately centred on the upward-facing punch face surface. The symbol for this mass is m_1. The use of a small amount of sticky sucrose solution or other adhesive between

the two parts, or some other method of keeping the infiltrant compact in place, shall be a matter of agreement between the parties concerned. The specimens shall not touch each other but shall be spaced well apart.

A reference infiltrating powder, which has been agreed upon by the parties concerned, shall be run at the same time as the infiltrant under test. Comparison between the reference and the tested infiltrant serves to verify that the test conditions, particularly furnace conditions and atmosphere, are not abnormal.

Extra skeleton specimens shall also be run through the infiltrating furnace cycle without an infiltrant compact in contact. After the furnace treatment, the mass of these extra specimens shall be determined (m_2). This value represents the mass of the original skeleton adjusted for the mass loss in the furnace treatment resulting from the reducible oxides, decarburization, loss of other volatiles, and the volatilization of lubricants when green skeleton specimens are used.

6.3 Specific infiltration conditions shall be a matter of agreement between the parties concerned. It is suggested that the following infiltration data be determined and recorded:

— heat-up time and rate, cooling time and rate, time at temperature;

— range of furnace temperatures during infiltration;

— furnace atmosphere, including moisture content at inlet to furnace or sampled from the hot zone, and the rate of flow with respect to the cross-section of the furnace hearth.

6.4 Observe and note the appearance of the infiltrated specimens as each tray is removed from the furnace.

Weigh each infiltrated specimen plus any residue, taking precautions to include the mass of any loose residue which may have fallen off. Any loose residue lying in the tray shall be assigned to this mass if it can be established that it has definitely come from this particular specimen and not from one of its neighbours. The mean mass of the infiltrated specimens is given the symbol m_3.

Remove all loose residue by inverting the infiltrated specimens and reweigh with the loose residue absent. In the case of a residue-free powder, the infiltrated specimens can be weighed as they are. Those infiltrants which produce a slightly adherent “button” will require a scraping action to remove this form of residue before weighing the specimen. The mean mass of the cleaned infiltrated specimens is given the symbol m_4.

Examine the infiltrated specimen surfaces, especially the one that has been in contact with the infiltrant compact, using approximately 10× magnification. Record the presence of adherent residue. Evaluate and note the amount of erosion. The different erosion ratings shall be established by agreement between the parties concerned. In addition to reporting erosion rankings, data shall also be reported as to the amount of infiltrant entry per unit area which produced that erosion rating. The amount of erosion tends to increase as the amount of infiltrant per unit area increases.

NOTE Generally, any infiltrant is considered unacceptable if there is any residue adherent to the infiltrated part. This can be determined by inspection as described above. If quantitative measures are desired, the amount of adhering residue can be determined by the difference in mass before and after removing any adherent residue by grinding or filing.

6.5 Accurately measure the dimensions of the infiltrated specimens.

The change in dimensions of the infiltrated test specimens may be determined by calculating the change in dimensions before and after infiltration and is expressed as a percentage. The dimensional change can also be reported as the change in dimensions after infiltration based on the die dimensions using the formulae given in ISO 4492.

6.6 Determine the density of the infiltrated specimen either by calculation from the mass and dimensions or by weighing in air and water as described in ISO 2738.

6.7 Determine the transverse rupture strength of the infiltrated specimens using the procedure described in ISO 3325 when the transverse rupture specimen is used (see 5.4).

6.8 Determine the hardness of the infiltrated specimens using the procedures described in ISO 4498. The location of the positions for hardness determination shall be a matter of agreement between the parties concerned.

7 Calculation and expression of results

7.1 Calculate the gross infiltration efficiency by the following formula:

$$\text{Efficiency, \%} = \frac{m_4 - m_2}{m_1} \times 100$$

where

m_4 is the mass of the infiltrated specimen, in grams, after residue has been removed;

m_2 is the mass of the sintered, uninfiltrated specimen, in grams (see 6.2);

m_1 is the mass of the infiltrant compact, in grams.

7.2 Calculate the loose residue amount as follows:

$$\text{Residue, \%} = \frac{m_3 - m_4}{m_1} \times 100$$

where

m_3 is the mass of the infiltrated specimen, plus all residue, in grams;

m_4 is the mass of the infiltrated specimen, in grams, after residue has been removed;

m_1 is the mass of the infiltrant compact, in grams.

8 Test report

The test report shall contain the following information:

a) composition, mass and density of the skeleton specimen;

b) identity or composition, mass and green density of the infiltrant used;

c) infiltrant loading density;

d) infiltration furnace conditions, including temperature, time, atmosphere and rate of flow;

e) appearance of infiltrated specimens;

f) erosion rating;

g) efficiency to the nearest 1 %;

h) residue amount to the nearest 1 % (anything less than 1 % is reported to the nearest 0,1 %);

i) dimensional change;

j) infiltrated density;

k) transverse rupture strength, if determined;

l) hardness.