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Permeable sintered metal materials — Determination of density, oil content, and open porosity

Matériaux métalliques frittés perméables — Détermination de la masse volumique, de la teneur en huile et de la porosité ouverte

Reference number
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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 2738 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy.*

It cancels and replaces International Standards ISO 2737 : 1973 and ISO 2738 : 1973, of which it constitutes a technical revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Permeable sintered metal materials — Determination of density, oil content, and open porosity

1 Scope and field of application

This International Standard specifies methods of determining the density, oil content, and open porosity of permeable sintered metal materials.

It applies in particular to porous metal bearings and to structural parts produced by pressing, and sintering metal powders.

2 References

ISO 758, *Liquid chemical products for industrial use — Determination of density at 20 °C.*

ISO 3448, *Industrial liquid lubricants — ISO viscosity classification.*

ISO 3507, *Pyknometers.*

ISO 4495, *Lubricated metallic powders — Determination of lubricant content — Soxhlet extraction method.*

3 Symbols and designations

Table 1

Symbol	Designation	Unit
m_1	Initial mass of the test piece	g
m_2	Mass of the test piece after oil extraction and drying	g
m_3	Mass of the fully impregnated test piece	g
m_4	Mass of the test piece and filled pyknometer with the test piece outside the pyknometer	g
m_5	Mass of the test piece and filled pyknometer with the test piece inside the pyknometer	g
m_a	Mass of the fully or partially impregnated test piece plus supporting device (for example suspension wire) weighed in air	g
m_w	Mass of the fully or partially impregnated test piece plus supporting device (for example suspension wire) weighed in water	g
V	Volume of the test piece	cm^3
ϱ_w	Density of the water used	g/cm^3
ϱ_1	Density of the oil initially in the test piece[1)]	g/cm^3
ϱ_2	Density of the impregnating oil used[1)]	g/cm^3
ϱ_L	Density of the liquid in the pyknometer	g/cm^3

1) The oil density is assumed to be known or, if not, to be determined according to ISO 758.

4 Principle

4.1 Density

The density of the test piece may be expressed in two ways.

4.1.1 Dry density

This is determined by dividing the mass after drying by the volume.

4.1.2 Fully impregnated density (wet density)

This is determined by dividing the fully impregnated mass by the volume.

NOTE — The volume of the fully impregnated test piece (i.e. the total volume including the pores) is determined by liquid displacement methods.

4.2 Oil content

The oil content of the test piece may be expressed in two ways.

4.2.1 As a percentage by volume

This is determined by dividing the volume of the oil by the volume of the test piece and multiplying the ratio by 100.

4.2.2 As a percentage of the volume of the open porosity

This is determined by dividing the volume of the oil by the volume of the open porosity and multiplying the ratio by 100.

4.3 Open porosity

The open porosity of the test piece is expressed as a percentage by volume, by dividing the oil content after full impregnation by the volume of the test piece and multiplying the ratio by 100.

4.4 Volume

The volume of the test piece is determined by weighing the test piece suspended in air, and then weighing the test piece immersed in a liquid of known density.

The volume is calculated as the difference between the two weighing results divided by the density of the liquid.

4.5 Determinations

Depending upon which of the properties is to be determined, some or all of the test procedures in clause 7 are carried out. Table 2 shows the test procedures that are carried out for the property to be determined. The values obtained are inserted in the respective formulae given in clause 8 to obtain the desired property.

5 Apparatus

5.1 Analytical balance, of sufficient capacity, and accurate to 0,01 %.

5.2 Soxhlet extractor, with oil solvent.

5.3 Device for weighing the test piece in air and in liquid.

The liquid is usually water (see figures 1, 2 and 3).

5.4 Vessel, large enough to accommodate the test piece and the device (5.3) for weighing it, containing distilled or deionized water, or preferably degassed water, with 1 or 2 drops of wetting agent added.

5.5 Apparatus for vacuum impregnation of the test piece with oil.

5.6 Impregnation oil, of known density (see ISO 758 for the determination of the density of liquids).

Table 2

Test procedure	Symbol for result obtained	Properties to be determined				
		Density		Oil content		Open Porosity
		Dry	Fully impregnated	% (V/V)	% of open porosity	
Initial weighing of the test piece (7.1)	m_1			X	X	
Extraction of the oil contained in the pores of the test piece (7.2)		X		X	X	X
Determination of the mass of the test piece after oil extraction and drying (7.3)	m_2	X		X	X	X
Full impregnation of the test piece with an oil of known density (7.4)			X		X	X
Determination of the mass of the fully impregnated test piece (7.5)	m_3		X		X	X
Determination of the volume of the test piece (7.6)	V	X	X	X		X

5.7 Pyknometer, of sufficient capacity, complying with the requirements of ISO 3507.

6 Test piece

6.1 If possible, the test piece shall be tested whole. If this is not possible, the test piece shall be cut or broken into smaller parts to facilitate the various operations; all the parts shall be subjected to the density determination. The density of the test piece shall be determined from the total mass and the total volume.

6.2 If the test piece is too small to be measured easily, either a number of test pieces shall be tested together to obtain the average value, or the pyknometer method shall be used (see 7.6.8).

NOTE — In general, this is the case when the individual pieces have a volume less than 0,5 cm^3.

6.3 The surface of the test piece shall be free of adhering dirt, grease, or other foreign material.

6.4 The surface of the test piece shall be free from surplus oil, for example that held by capillary action or surface tension. When removing any such surplus oil with an oil-absorbent material, care shall be taken to avoid removing oil contained in the pores.

NOTE — The presence of surplus oil on the surface of the test piece is most likely to occur after the full impregnation treatment.

7 Test procedures

7.1 Determination of the initial mass of the test piece

Weigh the test piece in the condition in which it was received, to obtain m_1.

NOTE — If the test piece is known to contain no oil, the procedures described in 7.2 and 7.3 are omitted. In this case, m_1 is substituted for m_2 in the formulae given in 8.1 and 8.3.

7.2 Removal of oil from the test piece by solvent extraction

Approximately 3 h of soaking and about 10 solvent changes are required to remove the oil from test pieces of average density and small wall thickness. For thick walls and high density, up to 24 h are sometimes required.

NOTE — The Soxhlet extractor is a convenient apparatus for soaking the test piece in warm freshly distilled oil solvent. The distillation rate determines the number of cycles and hence the number of solvent changes that occur.

A suitable Soxhlet unit is described in ISO 4495.

Continue the extraction to constant mass after evaporation of the solvent left in the pores.

NOTE — Experience will indicate the best extraction time and distillation rate to use.

Dry the test piece to constant mass (i.e. until the reduction in mass produced by the last extraction does not exceed 0,05 %) at a temperature of 20 °C above the boiling point of the solvent, then cool in a desiccator and weigh.

Choose the solvent so that complete dissolution of the oil is ensured. This requirement shall be tested for separately. The solvent used shall be stated in the test report.

For practical control purposes, other methods for removing the oil may be used (such as heating in a protective atmosphere). In cases of dispute, the Soxhlet extraction method shall be the reference method.

7.3 Determination of the mass of the dried test piece

Weigh the test piece after solvent extraction and drying to obtain m_2.

7.4 Impregnation with oil and surface coating

7.4.1 Full impregnation (for determination of the open porosity)

Submerge the test piece in oil, contained in any suitable vessel capable of withstanding a vacuum. Reduce the pressure on the surface of the oil to between 1 and 10 MPa.

Continue the vacuum treatment until no further bubbles appear on the surface of the oil.

Restore the pressure in the vacuum chamber to that of the ambient atmosphere. Allow the test piece to remain submerged in the oil for a period which is not less than the period of the vacuum treatment.

NOTE — For the majority of porous metals a single vacuum treatment is sufficient to ensure full impregnation. In some cases a second vacuum treatment is necessary to achieve full impregnation. This can be established by reducing the pressure a second time, and if no further air bubbles appear, it can be safely assumed that the first treatment has achieved full impregnation.

The oil shall be completely immiscible with water and shall wet the porous metal.

NOTE — Generally, the oil shall have a viscosity at 20 °C of between 50 and 500 mm^2/s* which is in the range ISO VG 22 to VG 150, as specified in ISO 3448. With a low-viscosity oil, impregnation is faster than with a high-viscosity oil.

Remove the test piece from the oil, allow to drain and remove the surplus surface oil as described in 6.4.

* 1 mm^2/s = 1 cSt

7.4.2 Partial impregnation (suitable for determination of the volume)

The requirements of the oil are the same as stated in 7.4.1.

Submerge the test piece in hot oil (65 ± 5 °C) until no further air bubbles appear. Cool the test piece to room temperature whilst still submerged in oil by removing it from the hot oil and quickly transferring it to cold oil. Remove the cooled test piece from the cold oil, allow to drain and remove the surplus surface oil as described in 6.4.

7.4.3 Surface coating methods (suitable for determination of the volume)

Coat the porous surface of the test piece with a film that prevents, by surface tension, the water from entering the pores.

The following techniques have been found to be suitable for particular types of porous metal. However, before any are used, the effectiveness of the technique with respect to the type and shape of porous metal must first be established.

7.4.3.1 Petroleum jelly

Smear the surface of the test piece with petroleum jelly and remove any excess.

7.4.3.2 Silicones

Many silicone fluids produce surface films which are not wetted by water. Dip the test piece into either the silicone fluid or a dilute solution of the silicone fluid in a suitable solvent and dry to constant mass.

7.4.3.3 Paraffin wax

Dip the test piece into a 5 % solution of paraffin wax in a suitable solvent and dry to constant mass.

7.5 Determination of the mass of the fully impregnated test piece

Weigh the test piece after full oil impregnation to obtain m_3.

7.6 Determination of the volume of the test piece

7.6.1 Determine the volume, V, of the test piece by weighing in air to obtain m_a, and then submerge in water or other liquid of known density to obtain m_w. Calculate the volume of the test piece by dividing the mass of displaced liquid by the density of the liquid.

7.6.2 With porous metals, it is essential that the liquid used is not absorbed by the pores. For this reason, the pores are impregnated with oil, and water is usually used as the test liquid, but it is not always necessary to impregnate a test piece fully. However, in order to ensure that no water enters the pores when the test piece is submerged in the water, the test piece may be partially impregnated or surface coated as described in 7.4.2 and 7.4.3. As a reference method, the test piece shall be fully impregnated with oil as described in 7.4.1.

NOTE — After weighing in water, the test piece should be reweighed in air (having removed any adhering water), to confirm that no water has been absorbed.

7.6.3 Figures 1, 2 and 3 show methods of suspending the test piece during weighing. In general, the mass and volume of the device should be as small as possible.

7.6.4 The test piece can be suspended from a piece of thin wire, and the total mass of the test piece and wire determined in air and in water. Allowance is made for the volume of the wire submerged in the water, but this is often insignificantly small when compared with the volume of the test piece. This allowance can be made by weighing the wire in air and then when submerged to the correct depth in water. Alternatively, the length of wire which is submerged can be measured, and the correction made based on the known volume of a unit length of wire.

7.6.5 Ensure that all air bubbles are removed from the surface of the test piece and the supporting device. It is permissible to add a few drops of wetting agent to the water. (See 5.4.)

7.6.6 The test piece and the water shall be at the same temperature. The normal test temperature is between 18 and 22 °C and the density, ϱ_w, of pure water in this range may be taken as 0,997 g/cm³. For temperatures outside this range, the water density shall be determined.

7.6.7 The volume, V, in cubic centimetres, is given by the equation

$$V = \frac{m_a - m_w}{\varrho_w}$$

7.6.8 The volume can be determined using a pyknometer filled with a liquid of known density ϱ_L. Impregnate the test piece fully with oil (see 7.4). Weigh the filled pyknometer together with the test piece outside the pyknometer, to obtain m_4.

Place the test piece in the pyknometer, ensuring that the test piece and the liquid are at the reference temperature as specified in ISO 3507. Remove any entrapped air by shaking. Top up the pyknometer with the liquid and replace the stopper. Remove all liquid from the outside of the pyknometer with an absorbent material, taking particular care over the region where the stopper and the neck meet. Leave until last the removal of the drop of liquid left on the stopper. Weigh the filled pyknometer, with the test piece inside it, to obtain m_5.

The volume, V, in cubic centimetres, is given by the equation

$$V = \frac{m_4 - m_5}{\varrho_L}$$

8 Expression of results

8.1 Density

The dry density, expressed in grams per cubic centimetre, is given by the formula

$$\frac{m_2}{V}$$

The fully impregnated density (wet density), expressed in grams per cubic centimetre, is given by the formula

$$\frac{m_3}{V}$$

Report the density to the nearest 0,01 g/cm³.

8.2 Oil content

The oil content, expressed as a percentage by volume, is given by the formula

$$\frac{m_1 - m_2}{\varrho_1 V} \times 100$$

Report the oil content to the nearest 0,1 % (V/V).

The oil content, expressed as a percentage of the open porosity, is given by the formula

$$\frac{m_1 - m_2}{\varrho_1} \times \frac{\varrho_2}{m_3 - m_2} \times 100$$

Report the oil content to the nearest 0,1 % in absolute value.

8.3 Open porosity

The open porosity, expressed as a percentage by volume, is given by the formula

$$\frac{m_3 - m_2}{\varrho_2 V} \times 100$$

Report the open porosity to the nearest 0,1 % (V/V).

9 Test report

The test report shall include the following information:

a) reference to this International Standard;

b) all details necessary for the identification of the test sample;

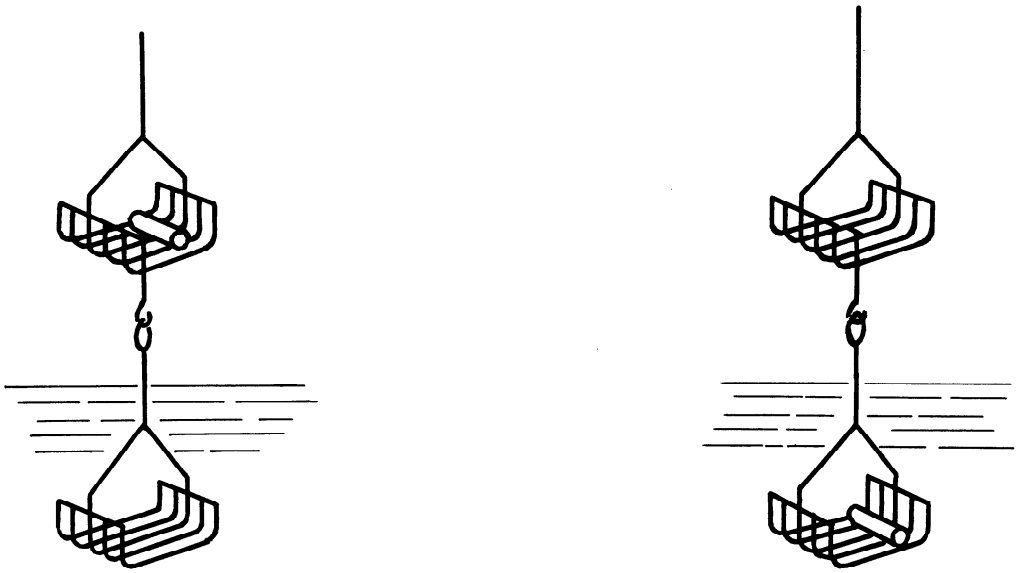
c) if the test piece has been subdivided, or, in the event that a number of test pieces have been tested together, the number;

d) the method used and the result obtained;

e) the value of the density of the oil initially present in the test piece, as well as the origin of this value (measured, known or assumed) in the case of determination of oil content;

f) all operations not specified by this International Standard, or regarded as optional;

g) details of any occurrence which may have affected the result.



a) Weighing in air

b) Weighing in water

Figure 1

a) Weighing in air

b) Weighing in water

Figure 2

a) Weighing in air

b) Weighing in water

Figure 3

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