INTERNATIONAL STANDARD

ISO 4003

Permeable sintered metal materials — Determination of bubble test pore size

Matériaux en métal fritté perméable — Détermination de la dimension des pores — Méthode bulloscopique

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FOREWORD

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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.

International Standard ISO 4003 was developed by Technical Committee ISO/TC 119, *Powder metallurgical materials and products,* and was circulated to the member bodies in December 1975.

It has been approved by the member bodies of the following countries :

Australia	Japan	Spain
Austria	Mexico	Sweden
Canada	Poland	United Kingdom
France	Portugal	U.S.A.
Germany	Romania	U.S.S.R.
Italy	South Africa, Rep. of	Yugoslavia

No member body expressed disapproval of the document.

Permeable sintered metal materials — Determination of bubble test pore size

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method, known as the bubble test method, for the determination of the pore size of permeable sintered powder metallurgical materials, i.e. filters, porous bearings, porous electrodes and other parts with interconnected porosity.

NOTE — The bubble test shall be considered as a quality control test and not as a test for defining filter grades or determining exact pore size and pore size distribution.

2 REFERENCE

ISO 2738, *Permeable sintered metal materials — Determination of density and open porosity.*

3 PRINCIPLE

Impregnation of a test piece with a test liquid. Immersion of the test piece in the test liquid and introduction of a gas (usually air) into the test piece at gradually increasing pressure. Determination of the pressure at which bubbles are emitted from the surface of the test piece. Evaluation of the equivalent bubble test pore size by means of a mathematical formula.

4 DEFINITION

bubble test pore size : The maximum equivalent capillary diameter in the test piece which is calculated from the measured minimum pressure required to force the first bubble of gas through the test piece (under standardized conditions) impregnated with a liquid.

The first bubble of gas will form at the pore having the greatest throat, the throat being the narrowest section of this pore.

For calculation purposes, it is assumed that this bubble forms at the end of a capillary tube of circular cross-section which is initially filled with the same liquid of known surface tension.

For a circular capillary, the diameter is related to the bubble pressure by the equation :

$$d = \frac{4\,\gamma}{\Delta p} \qquad \ldots (1)$$

where

d is the capillary diameter corresponding to the bubble test pore size, in metres;

γ is the surface tension of the test liquid, in newtons per metre;

Δp is the differential pressure, in pascals, across the test piece under static conditions, i.e.

$$\Delta p = p_g - p_l \qquad \ldots (2)$$

p_g being the gas pressure, in pascals;

p_l being the pressure in the liquid at the level of bubble formation, in pascals :

$$p_l = 9{,}81 \times \rho_l \times h \qquad \ldots (3)$$

where ρ_l is the density of the test liquid, in kilograms per cubic metre;

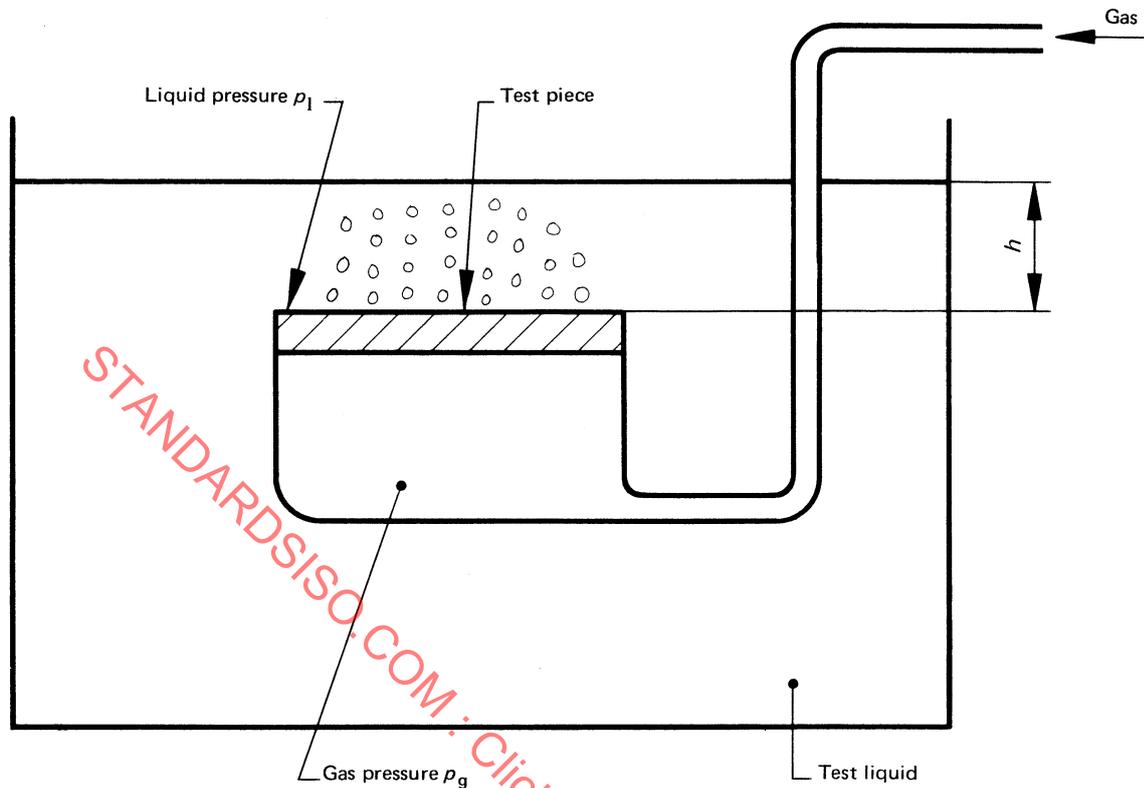h is the height of the surface of the test liquid, in metres, above the highest throat in the test piece.

FIGURE — **Bubble test assembly**

NOTES

1 The bubble test pore size corresponds to the minimum differential pressure at which constant bubbling first occurs. For this reason, this pressure is sometimes termed "minimum bubble pressure" or "first bubble point". The corresponding capillary diameter is sometimes termed "maximum pore size" or "maximum pore diameter", or "largest pore size". However, the maximum pore size determined with this method may be the result of a single local defect, and therefore not representative of the pore population.

2 As the gas pressure increases beyond the minimum bubble pressure (first bubble point), different aspects of bubbling occur on the test piece. The pressure for a given aspect can lead to the definition of a conventional pore size. For example, the pressure for which a generalized bubbling occurs is frequently specified (foaming over the whole surface). These particular definitions should be agreed between the supplier and the user. Moreover, the uniformity of distribution of pores approaching the maximum pore size may be observed by gradually increasing the gas pressure. Cracks and clogged areas are easily discerned by this operation.

3 The bubble test does not constitute a measurement of the maximum size of particle that the permeable test piece will pass (retentivity of a filter). A filter may be expected to retain all particles larger than the maximum pore size as defined by the bubble test; but, because of irregularity in shape of pores and other phenomena related to the filtration process, the same filter will retain particles which are much smaller than the maximum pore size. The determination of the size of the largest non-deforming particle which can pass through the porosity requires time-consuming methods, for example a glass-bead test. For estimation purposes it is useful to take empirical factors, which are to be multiplied with the bubble test pore size calculated from equation (1). The factor is about 0,4 for porous metal made from uniform spherical particles; and about 0,2 for porous metal made from irregular particles.

5 APPARATUS

5.1 Dry and filtered gas (generally air) supply, at an adequate pressure.

5.2 Pressure regulator, affording constant and precise control of the gas pressure, i.e. a gradual increase of the pressure at a predetermined rate, or a stepwise increase of pressure and the facility to maintain a constant pressure at each step.

5.3 Flow-meter, if required.

5.4 Device to measure the effective gas pressure, measuring to an accuracy of $\pm 1\,\%$ (mechanical pressure gauge, or water or mercury manometer). This device shall be placed close to the bubble test cell, in order to facilitate the simultaneous observation of the bubble appearance and the pressure value.

5.5 Assembly for observing the bubble appearance at the surface of the test piece, according to the shape of the test piece, and for ensuring that the test piece is completely saturated with the liquid, and immersed under a constant depth of the liquid throughout the test. If the test piece is hollow or of a shape other than flat, it should be rotated with its major axis horizontal so that the whole surface is examined.

5.6 Test liquid, selected in relation to the kind of metal composing the test piece. Among pure liquids which wet metals perfectly, 95 % ethanol, methanol, isopropanol or carbon tetrachloride are most generally employed (see the table for properties). The test is carried out at room temperature (20 ± 5 °C). The surface tension, γ, of the test liquid may be obtained from tables of physical constants.

TABLE — **Test liquids suitable for use with permeable metals**

Test liquid	Density g/cm^3	Surface tension at 20 °C N/m
Methanol	0,79	0,022 5
Ethanol, 95 %	0,805	0,023
Isopropanol	0,79	0,021 5
Carbon tetrachloride[1]	1,59	0,027

1) As carbon tetrachloride vapour can be dangerous to health, the usual laboratory precautions should be taken.

6 PROCEDURE

The test piece shall be clean, dry and free from extraneous material and any trace of grease or similar substances likely to hinder the perfect and uniform wetting action of the test liquid.

Impregnate the test piece completely with the test liquid. Insert it in the bubble test apparatus and maintain it fixed, immersed under the smallest depth of test liquid consistent with the convenient observation of the appearance of the bubbles. Measure this depth h (see the figure) and the temperature of the liquid.

From an effective gas pressure of zero, increase the pressure regularly at a rate of between 20 and 100 Pa/s (according to the estimated pore size), while the surface of the test piece is under constant observation. In the case of a hollow cylindrical test piece, rotate it so that all of the surface is observed; in this case, increase the pressure in steps, each of from 50 to 500 Pa (according to the estimated pore size), so that each new pressure value is maintained whilst the surface is observed during the rotation.

Note the first bubble pressure when a string of bubbles occurs from one distinct point (or perhaps several distinct points at the same time).

If there is a defect, the first bubble may appear in a zone remote from the upper surface. In this event, reduce the pressure and repeat the test after rotating the sample on its own axis; increase the pressure again incrementally.

NOTES

1 The test piece shall be impregnated to ensure the saturation of its open porosity. A vacuum impregnation in accordance with the requirements given in ISO 2738 is recommended.

2 When a determination is repeated on the same sample, it is necessary to re-impregnate the sample totally before retesting.

3 It is important that the test piece be properly sealed in the apparatus. If bubbles emerge from the proximity of the seals, the result shall be ignored and the test repeated with improved seals.

4 When the test piece is composed of a layer of fine pores supported on a base of coarser pores, the bubble test pore size characterizing the material is that of the fine pore layer. The test piece shall be tested in such a way that the bubbles appear at the surface of the fine pore layer.

5 By agreement between user and supplier, pore sizes corresponding to other bubbling conditions may be determined as indicated in clause 4, note 2.

7 EXPRESSION OF RESULTS

Calculate the bubble test pore size, using the formula in clause 4. Report the arithmetical mean of three determinations rounded to the nearest 5 %.

8 TEST REPORT

The test report shall include the following information :

a)   reference to this International Standard;

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

c)   the liquid used;

d)   the rate of pressure increase;

e)   the position of the first bubble;

f)   the result obtained;

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

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

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