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Metallic powders — Determination of envelope-specific surface area from measurements of the permeability to air of a powder bed under steady-state flow conditions

Poudres métalliques — Détermination de la surface spécifique d'enveloppe à partir de mesures de la perméabilité à l'air d'un lit de poudre dans des conditions d'écoulement permanent

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Foreword

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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).

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

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This second edition cancels and replaces the first edition (ISO 10070:1991), which has been technically revised.

The main changes compared to the previous edition are as follows:

— introduction of an automated test device based on the Gooden and Smith method, including procedure and calibration.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Introduction

The measurement of the permeability of a packed powder bed to a laminar gas flow is the basis of this document. The determination can be made either at constant pressure drop (steady-state flow) or at variable pressure drop (constant volume). This document deals only with determinations made under steady-state flow conditions.

The permeability measured is influenced by the porosity of the powder bed. For a given particle shape, the values of permeability and porosity can be used to calculate a specific surface area of the powder by means of different formulae.

The surface area so calculated includes only those walls of the pores in the powder bed which are swept by the gas flow. It does not take into account closed or blind pores. It is known as the envelope-specific surface area. It can be very different from the total surface area of particles as measured, for instance, by gas adsorption methods.

A single equation is used in the standard methods described in this document. It entails certain limitations with respect to the type of powder (particle shape) and the porosity of the powder bed for which the method is applicable. Consequently, this is not an absolute method, and the value obtained depends upon the procedure used and the assumptions made.

The specific surface area determined can be converted into a mean equivalent spherical diameter (see Clause 3).

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Metallic powders — Determination of envelope-specific surface area from measurements of the permeability to air of a powder bed under steady-state flow conditions

1 Scope

This document specifies a method of measuring the air permeability and the porosity of a packed bed of metal powder, and of deriving therefrom the value of the envelope-specific surface area. The permeability is determined under steady-state flow conditions, using a laminar flow of air at a pressure near atmospheric. This document does not include the measurement of permeability by a constant volume method.

Several different methods have been proposed for this determination, and several test devices are available commercially. They give similar, reproducible results, provided that the general instructions given in this document are respected, and the test parameters are identical.

This document does not specify a particular commercial test device and corresponding test procedure. However, for the convenience of the user, an informative annex has been included (see Annex A) which is intended to give some practical information on three specific methods:

— the Lea and Nurse method, involving a test device which can be built in a laboratory (see A.1);

— the Zhang Ruifu method, using a similar test device (see A.2);

— the Gooden and Smith method, involving a test device which can be built in a laboratory but for which a commercial test device also exists. (Two types of commercial test device exist; one of these is no longer available for purchase, but is still being used, see A.3.)

These methods are given as examples only. Other test devices available in various countries are acceptable within the scope of this document.

This testing method is applicable to all metallic powders, including powders for hardmetals, up to 1 000 µm in diameter, but it is generally used for particles having diameters between 0,2 µm and 75,0 µm. It is not intended to be used for powders composed of particles whose shape is far from equiaxial, i.e. flakes or fibres, unless specifically agreed upon between the parties concerned.

This testing method is not applicable to mixtures of different metallic powders or powders containing binders or lubricant.

If the powder contains agglomerates, the measured surface area can be affected by the degree of agglomeration. If the powder is subjected to a de-agglomeration treatment (see Annex B), the method used is to be agreed upon between the parties concerned.

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 3954, *Powders for powder metallurgical purposes — Sampling*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
envelope density
mass of a powder bed divided by its *envelope volume* (3.3)

Note 1 to entry: The envelope density may be less than the solid density when particles contain pores that do not contribute to the gas flow through the powder bed.

3.2
envelope-specific surface area
specific surface area of a powder as determined by gas permeametry

3.3
envelope volume
volume occupied by the particles in a powder bed, excluding the volume of the *interstices* (3.5)

Note 1 to entry: In permeametry, the envelope volume comprises the volume of the solid matter plus the volume of all the pores which do not contribute to gas flow (closed pores, blind pores, micropores, surface micropores, surface roughness, etc.). Since this volume cannot be measured by any known method, it is taken, for the purposes of this document, as being equal to the effective volume, as determined by pyknometry.

3.4
equivalent sphere diameter
diameter of theoretical non-porous spherical particles of identical size, with which the same method of permeametry as that used for the powder under examination would give the same *volume-specific surface area* (3.9)

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3.5
interstices
spaces between particles in a powder bed, through which the air flows

3.6
mass-specific surface area
surface area of a powder divided by its mass

Note 1 to entry: This area depends on the type of method used for its determination.

3.7
permeability
ability of a porous material to allow a fluid to flow through it

Note 1 to entry: to entry In this document, the fluid used is dry air.

3.8
permeable porosity
volume of *interstices* (3.5) divided by the volume of the powder bed

3.9
volume-specific surface area
surface area of a powder divided by its effective volume (i.e. by its envelope volume)

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4 Symbols

Table 1 — Symbols used in the text

Symbol	Meaning	Unit	Observations
	Powder bed		
A	Cross-sectional area	m^2	Area of whole cross-section of powder bed perpendicular to flow direction: $A = \frac{\pi d^2}{4}$
d	Diameter of measuring cell	m	
L	Thickness (or height)	m	
m	Mass of powder	kg	
ϱ_e	Envelope density	kg/m^3	
ϱ	Solid density	kg/m^3	
ε_p	Permeable porosity		$\varepsilon_p = 1 - \frac{m}{AL\varrho_e}$
ε	Total porosity		$\varepsilon = 1 - \frac{m}{AL\varrho}$
	Gas flow		
q	Volume flow rate	m^3/s	Converted to standard conditions (STP - 0 °C, 1 atm)
p	Mean gas pressure	N/m^2	
Δp	Pressure drop	N/m^2	
η	Viscosity of gas	Ns/m^2	
T	Temperature of gas	K	
M	Molar mass of gas	kg/mol	M = 0,029 kg/mol for air
R	Molar gas constant	$\frac{J}{mol\,K}$	$R = 8{,}31 \frac{J}{mol\,K}$
	Calculation		
K	Kozeny-Carman factor		For the purposes of this document, $K = 5{,}0$
δK_0	Compound constant		For the purposes of this document, the generally accepted value of 2,25 is used
S_w	Mass-specific surface area	m^2/kg	
S_K	Kozeny term	m^{-1}	Formula (3)
S_m	Slip flow term	m^{-1}	Formula (4)
S_V	Volume-specific surface area	m^{-1}	$S_V = \varrho_e S_w$
Φ	Permeability	m^2	
D	Equivalent sphere diameter	m	$D = \frac{6}{S_V} = \frac{6}{\varrho_e S_w}$

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5 General principles

5.1 Permeability

Basically, permeametry is the experimental determination of the permeability, Φ, of a powder bed, the porosity of which is known.

The permeability is determined by measuring the volume flow rate, q, and the drop-in pressure, Δp, of a dry gas (generally air) continuously traversing the powder bed under laminar flow conditions.

The permeability is then calculated from Darcy's law, as shown in Formula (1):

$$\Phi = \frac{q\eta L}{A\,\Delta p} \tag{1}$$

5.2 Carman-Arnell and Kozeny-Carman formulae

The Carman-Arnell formula, as shown in Formula (2), relates specific surface area to the porosity and permeability of a packed bed of powder and takes into account both the viscous flow and the slip flow. This formula can be written as:

$$\Phi = \frac{\varepsilon_p}{K\eta}\left[\frac{\varepsilon_p^2}{S_V^2\left(1-\varepsilon_p\right)^2} + \frac{8}{3}\sqrt{\frac{2RT}{\pi M}} \times \frac{\delta K_0 \eta \varepsilon_p}{pS_V\left(1-\varepsilon_p\right)}\right] \tag{2}$$

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The solution of Formula (2), which is quadratic in S_V, can be simplified by calculating the value of two terms, the Kozeny term S_K and the slip flow term S_m, and then combining them to give S_V.

The Kozeny term S_K is given by Formula (3):

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$$S_K = \sqrt{\frac{A\,\Delta p \varepsilon_p^3}{K\left(1-\varepsilon_p\right)^2 L\eta q}} \tag{3}$$

The Kozeny term is identical to the Kozeny-Carman formula for S_V and gives the contribution to the surface area of the powder due to streamline flow.

The slip flow term S_m is given by Formula (4)

$$S_m = \frac{A\,\Delta p}{KLq} \times \frac{8}{3}\sqrt{\frac{2RT}{\pi M}} \times \frac{\delta K_0 \varepsilon_p^2}{p\left(1-\varepsilon_p\right)} \tag{4}$$

or, in the case of air, Formula (5):

$$S_m = 81 \times S_K^2 \frac{\left(1-\varepsilon_p\right)\eta}{p\,\varepsilon_p}\sqrt{T} \tag{5}$$

S_v is then given by Formula (6):

$$S_V = \frac{S_m}{2} + \sqrt{\frac{S_m^2}{4} + S_K^2} \tag{6}$$

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and the mass-specific surface area S_w by Formula (7):

$$S_w = \frac{S_V}{\varrho_e} \tag{7}$$

The equivalent sphere diameter D is given by Formula (8):

$$D = \frac{6}{S_V} = \frac{6}{\varrho_e S_w} \tag{8}$$

The Carman-Arnell formula, Formula (2), shall be used when the volume-specific surface area is greater than 10^6 m^{-1} (mean particle size less than 6 µm), because the slip flow component of the permeability becomes significant in addition to the viscous flow term.

For coarser powders, the Kozeny-Carman formula, Formula (3), may be used by agreement between the parties concerned; the error introduced by neglecting slip flow is about 10 % at a mean particle size of 6 µm and increases as the powder becomes finer.

The mass-specific surface area, S_w, is given by Formula (9):

$$S_w = \sqrt{\frac{\varepsilon_p^3 A \Delta p}{K\left(1-\varepsilon_p\right)^2 q \eta L \varrho_e^2}} \tag{9}$$

5.3 General

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The methods and test devices used in practice differ depending on the way in which the volume flow rate of the gas and the pressure drop are measured. Annex A describes three methods by way of example. The Kozeny-Carman relation applies only over a limited range of powder bed porosities, the range depending on the type of powder. It applies best to equiaxial powders. The Kozeny factor K varies with the particle shape and particle size distribution. In this document, the value of K is taken to be 5,0 but other values may be used by agreement between the parties concerned.

Due to the limitations of the Kozeny-Carman relation, the variation of the specific surface area as a function of porosity shall first be determined experimentally for any particular type of powder.

For example, make several successive determinations of the permeability, using test portions of the same mass from the same laboratory sample, and packing the powder bed to give a decreasing series of porosities. Over a certain range of porosities, the specific surface area will be practically constant. Only determinations made within this range shall be taken as valid.

5.4 Envelope density

In Formulae (1) to (9), the permeable porosity ε_p of the powder bed and the envelope density ϱ_e of the particles are used. They are related by Formula (10):

$$\varepsilon_p = 1 - \frac{m}{AL\varrho_e} \tag{10}$$

The envelope density ϱ_e is equal to the solid density only for smooth, non-porous particles. In such cases, $\varepsilon_p = \varepsilon$.

In all other cases, the envelope density ϱ_e shall be measured by an appropriate pyknometric method. The solid density value ϱ, or another density, may be adopted instead of the envelope density by agreement between the parties concerned.

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