INTERNATIONAL STANDARD

ISO 10076

First edition
1991-12-15

Metallic powders — Determination of particle size distribution by gravitational sedimentation in a liquid and attenuation measurement

Poudres métalliques — Détermination de la distribution granulométrique par sédimentation par gravité dans un liquide et mesure de l'atténuation

Reference number
ISO 10076:1991(E)

Foreword

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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.

International Standard ISO 10076 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*.

Annexes A, B and C of this International Standard are for information only.

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Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Introduction

The settling behaviour under gravity of a given mass of particles dispersed in an initially static liquid is the basis of widely used sedimentation techniques for particle-size determination. The particle size is determined from the settling velocity by the use of Stokes' equation. The particle diameter so determined, the Stokes diameter, is the diameter of a sphere having the same density and the same free-fall velocity as the particle in a fluid of a given density and viscosity. The particle concentration must be low so that interaction between particles is negligible, and the Reynolds number must be low so that laminar flow conditions prevail.

Monitoring of the concentration of particles at a known depth below the surface of an initially homogeneous suspension enables the particle-size distribution to be calculated as a function of the measured surface or mass.

In this International Standard, two attenuation methods for the determination of concentration are considered:

— absorption of a beam of light;

— absorption of a beam of X-rays.

Although they are indirect, these sedimentation-attenuation methods are frequently employed in powder metallurgy. They give reproducible results as long as precise conditions of preparation of the suspension and of measurement are followed.

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Metallic powders — Determination of particle size distribution by gravitational sedimentation in a liquid and attenuation measurement

1 Scope

This International Standard specifies methods for determining the particle size distribution of metallic powders by measuring the attenuation of an electromagnetic beam projected through a suspension of particles settling in a liquid under gravity.

The methods are suitable only where Stokes' equation is applicable, i.e. laminar flow corresponding to a Reynolds number less than 0,25, and for particles whose settling rate is not affected by Brownian motion. They are therefore suitable for all metallic powders, including powders for hardmetals, containing particles in the size range 1 µm to 100 µm. They should not, however, be used for

a) powders containing particles whose shape is far from equiaxial, i.e. flakes or fibres, except by special agreement;

b) mixtures of powders;

c) powders containing lubricant or binder;

d) powders which cannot be dispersed in a liquid.

These considerations set an upper and a lower limit on the size of particles to be tested by a sedimentation method (see 5.1).

If the largest particle present in the sample exceeds this limit, the viscosity of the liquid needs to be increased to meet this requirement.

Stokes' law may be assumed to be valid for an initial concentration of the powder in the liquid of up to 0,5 % (V/V). In some cases, higher concentrations of up to 1 % (V/V) still give correct results, but validation tests are necessary for each material.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3252:1982, *Powder metallurgy — Vocabulary.*

ISO 3954:1977, *Powders for powder metallurgical purposes — Sampling.*

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 Stokes diameter: Diameter of a sphere having the same density and the same free-fall velocity as a powder particle in a fluid of a given density and viscosity.

3.2 effective density: Ratio of the mass of the powder to the volume as measured by pyknometry.

3.3 sedimentation height: Vertical distance between the upper surface of the suspension and the level of measurement of concentration.

3.4 cumulative undersize by mass: Mass of all the particles whose Stokes diameter is less than a given value. This is expressed as a percentage of the total mass of the particles.

3.5 mass fraction: Mass of all the particles whose Stokes diameter lies between two given values. This

mass fraction is expressed as a percentage of the total mass of the particles.

3.6 blank intensity: Intensity of the emergent beam with clear liquid in the sedimentation cell.

3.7 suspension intensity: Intensity of the emergent beam during sedimentation.

4 Symbols

Table 1 — Meanings of the symbols used in the text

Symbol	Meaning	Unit	Observations
Sedimentation system			
g	Acceleration due to gravity	m/s^2	$g = 9{,}81\ m/s^2$
L	Absorption length	m	Length of the light or X-ray beam path through the suspension
h	Sedimentation height	m	
c_0	Initial concentration	kg/m^3	
c	Concentration	kg/m^3	
ϱ_1	Density of the liquid	kg/m^3	
ϱ_e	Effective density of the powder	kg/m^3	
η	Viscosity of the liquid	$\frac{N \cdot s}{m^2}$	
Measurement			
I_0	Blank intensity		
I	Suspension intensity		
D	Optical density		$D = \log_{10}\left(\frac{I_0}{I}\right)$
K_m	Extinction coefficient		
A_w	Mass projected area of the particles in random orientation	m^2/kg	
S_w	Mass-specific surface area of the powder	m^2/kg	
μ_a	Mass absorption coefficient of the atoms present in the powder		
t	Sedimentation time	s	
d_{st}	Stokes diameter	m	
ΔM	Mass of particles between two given diameters	kg	
q	Mass fraction	%	$q = \frac{\Delta M}{\sum_{d=0}^{d=d_m} \Delta M} \times 100$
Q	Cumulative undersize by mass	%	$Q = \frac{\sum_{d=0}^{d=d_{st}} \Delta M}{\sum_{d=0}^{d=d_m} \Delta M} \times 100$
d_m	Stokes diameter of the largest particle in the suspension	m	

5 Principle

A horizontal beam of parallel light or X-rays is directed through a suspension of a powder in a liquid, at a known depth h below the surface (see figure 1).

If the suspension is assumed to be initially homogeneous, with concentration c_0 at time $t = 0$, and the particles are then allowed to settle under gravity, the number of particles leaving the level of the beam will initially be balanced by the number entering it from above and no change of concentration will be recorded. When the largest particle present at the surface of the suspension (diameter d_m) has fallen from the surface to the measurement level, there will be no similar particle entering the measurement level to replace it, and the concentration at this level will then begin to decrease. Hence the concentration c of particles present at depth h and time t will be the concentration of particles smaller than d_{st}, where d_{st} is given by Stokes' equation relating the Stokes diameter d_{st} and steady-state velocity $v = h/t$.

$$d_{st} = \sqrt{\frac{18\eta h}{(\varrho_e - \varrho_1)gt}} \qquad \ldots (1)$$

The mechanism of attenuation of the beam is different for visible light and for X-rays. For visible light, an indirect relationship between the surface area of the powder and its optical density applies. For X-rays, the measured concentration is directly proportional to the cumulative undersize by mass Q.

5.1 Size limit

The Reynolds number is defined as

$$R_e = \frac{\varrho_1 v d_{st}}{\eta} \qquad \ldots (2)$$

Combining equations (1) and (2) and applying the condition $R_e < 0{,}25$ gives the size limit

$$(d_{st})_{max} = \sqrt[3]{\frac{4{,}5\eta^2}{\varrho_1 g(\varrho_e - \varrho_1)}}$$

For example, for bronze ($\varrho_e = 8\,900$ kg/m^3) sedimenting in water ($\eta = 0{,}001$ N·s/m^2; $\varrho_1 = 1\,000$ kg/m^3), the upper particle-size limit is about 40 µm.

The mean displacement of submicron particles due to Brownian motion often exceeds the settling distance. In addition, fine particles may require very long sedimentation times or small sedimentation heights. The former are not practical, however, and the latter lead to poor resolution of the distribution curve. For these reasons, sedimentation under gravity is only recommended down to a Stokes diameter of about 0,5 µm for bronze and iron, for example, and down to a Stokes diameter of 1 µm for metals of lower density.

5.2 Particle density

In the case of porous or spongy particles, it is difficult to define correctly their effective density when sedimenting. Open pores may be partially filled by liquid while closed pores will be empty, and the effective particle density ϱ_e in Stokes' equation is less than the solid density.

A pyknometric measurement of density in a suitable liquid may be required; it gives a value closer to the effective density than the assumed solid density.

In any case, the density value adopted for calculation of Stokes diameters shall be stated.

5.3 Light absorption (photosedimentation)

Making a number of simplifying assumptions, the absorption of light by a suspension can be described by the Lambert-Beer law:

$$\ln(I_0/I) = A_w c L K_m \qquad \ldots (3)$$

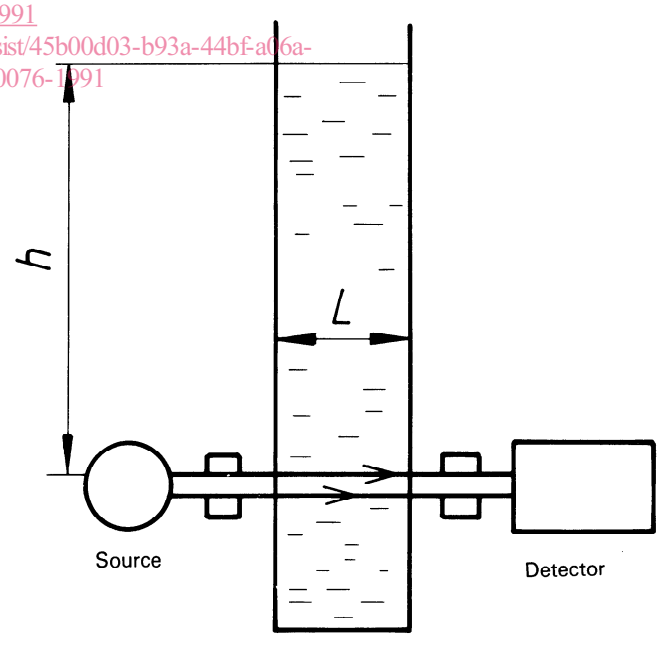

Figure 1 -- General arrangement of sedimentation apparatus

The following assumptions are usually made

- the particles are opaque and convex in shape (to satisfy the theory that the external surface is equal to 4 times the projected surface in random motion);
- the initial optical density is less than 0,7 (to give good resolution);
- the extinction coefficient is constant (generally K_m is assumed to be equal to unity).

Equation (3) may then be written

$$\ln (I_0/I) = S_w c L K_m/4 \quad \ldots (4)$$

$$= S_w c L/4 \quad \text{if } K_m \text{ is assumed to be 1.}$$

It should be noted that the above assumptions are not in fact valid, since K_m is dependent on the particle size, the particle-size distribution and the ability of the particles to transmit light. The attenuation is therefore not directly related to S_w and the method can therefore only be used to compare similar powders. K_m also depends upon the geometry of the light beam, and hence it is necessary to specify the instrument used.

The measured optical density is proportional to the cumulative proportion of undersize particles by surface. This may be converted to a mass distribution either by calculation based on the incremental form of equation (4), or by graphical summation (see worked example in annex A, clause A.1).

Equation (4) can be written in terms of the optical density D:

$$D = 0{,}434 \times \frac{A}{4s} \quad \text{or} \quad D = \alpha A \quad \ldots (5)$$

where

A is the surface area of all the particles present in the volume defined by a light beam of cross-section s;

α is a proportionality constant.

Consider now the change of state of the suspension at the beam level during a short interval of time $\Delta t = t_2 - t_1$, t_1 and t_2 being related to Stokes diameters d_1 and d_2 by equation (1). Between times t_1 and t_2, the particles belonging to the fraction limited by diameters d_1 and d_2 will disappear from the beam zone. Their mass is, by definition, ΔM. The total surface area of the particles present in the beam will decrease by an amount ΔA (the surface area of the particles which disappear from that level) which is given by

$$\Delta A = S_{1-2} \times \Delta M \quad \ldots (6)$$

$$= \frac{6}{\varrho_e d} \times \Delta M$$

where S_{1-2} is the mass-specific surface of this fraction of particles, which are assumed to have a mean Stokes diameter $\bar{d}$ given by

$$\bar{d} = \frac{d_1 + d_2}{2} = \frac{6}{\varrho_e S_{1-2}}$$

Rearrangement of equation (6) gives

$$\Delta M = \frac{\varrho_e}{6} \bar{d} \Delta A$$

From equation (5), the change in measured optical density is

$$\Delta D = \alpha \Delta A$$

Finally, the equation to be used when calculating the mass distribution for any size interval d_1 to d_2 is

$$\Delta M = \beta \bar{d}\, \Delta D \quad \ldots (7)$$

where β is a constant.

5.4 X-ray absorption (X-ray sedimentation)

With a continuous spectrum of X-rays, the absorption is proportional to the mass of powder present in the beam. Thus

$$\ln \left(\frac{I_0}{I} \right) = \mu_a c L$$

The concentration is directly proportional to the cumulative undersize by mass. Thus

$$\frac{Q}{100} = \frac{c}{c_0}$$

6 Procedure

6.1 Preparation of the test sample

The powder to be tested is taken in the as-delivered state and sampled in accordance with ISO 3954. Neither disintegration, nor milling nor any other treatment of the powder is allowed, except by agreement between the interested parties. In the latter case, a detailed description of the treatment used shall be drawn up, or a reference to such a description given. Annex B gives, as examples, some methods for de-agglomeration of the sample.

When the powder to be tested contains an appreciable proportion of coarse particles (i.e. diameter greater than 100 µm), these particles shall be separated by sieving before the sedimentation test. Use of this procedure shall be reported.

6.2 Preparation of the suspension

6.2.1 Selection of the suspending liquid

The liquid used to make the suspension shall satisfy the following requirements:

— the density of the liquid shall be lower than the density of the solid;

— the solid shall not dissolve in or react with the liquid;

— the liquid shall wet the solid so that agglomerates are not formed;

— the viscosity of the liquid shall be such that the test does not take too long and the coarsest particles do not settle too rapidly (see 6.1).

Frequently, a pure liquid does not produce a good dispersion of the particles. In such cases, a dispersing or wetting agent shall be used, either dissolved in the liquid or added to the powder.

Annex C gives, as examples, a list of suspending liquids and dispersing agents. The suitability of a particular system shall be checked as indicated in 6.2.2 below.

6.2.2 Tests for dispersion

Several methods can be used to test whether a suspension is free from agglomerates.

6.2.2.1 Microscopic examination

A drop of the prepared suspension is placed on a microscope slide, and a cover glass is lowered with extreme care over it. Examination of the preparation under a suitable magnification will show if the particles are entirely separate and form a good dispersion, or are assembled in chains or clusters.

6.2.2.2 Qualitative sedimentation

The suspension is left to settle. A correctly dispersed suspension settles less rapidly than a flocculated suspension and shows no sharp boundary between the clear liquid and the turbid layer as settling proceeds. The sediment produced is rigid and compact and has a minimum volume.

6.2.2.3 Quantitative sedimentation with photometric measurement

Attenuation measurements are made immediately after agitation using different suspending liquids and the same powder concentration. A maximum value of optical density indicates an optimum dispersion.

6.2.2.4 Quantitative sedimentation with X-ray absorption

Quantitative tests are made with suspensions in different liquids and having different concentrations, starting for instance with a low concentration of 0,1 % (V/V). The best system is the one which gives the highest proportion of fine fractions.

6.2.3 Preparation of the suspension

6.2.3.1 The density and viscosity of the selected liquid at the test temperature shall be known or measured. The suspension may be prepared at the selected concentration, starting with a weighed test portion, or the suspending liquid may be added slowly and the powder worked to a paste, then diluted to a suspension. A drop of wetting agent shall be added to the dry test portion or the suspending liquid if the liquid does not readily wet the powder. Make up to an appropriate volume.

6.2.3.2 The dispersion may be made by shaking the suspension or stirring it in the sedimentation vessel. If necessary, treat with ultrasonics or de-aerate under reduced pressure, either in a separate vessel or in the sedimentation vessel. The duration and intensity of dispersion is selected so that unwanted agglomerates are destroyed but any aggregates or desired particles are not damaged.

6.3 Sedimentation test

6.3.1 The general requirements for a satisfactory test are the following:

— the sedimentation cell shall be installed rigidly in a vertical position and without vibration;

— the cell shall be fitted with a thermostatic envelope, or the environment conditioned to maintain the cell at a known temperature, stable to within ± 1 °C, with a rate of change of temperature of less than 0,01 °C/min for prolonged analyses (longer than 1 h);

— care shall be taken to avoid convection currents in the liquid (due to evaporation or to external heat sources, for instance) and mass transfer currents (due to density inversion in the suspension).

6.3.2 Stir the suspension using either a stirring rod or a magnetic bar until the analysis is started ($t = 0$).

6.3.3 In most instruments, the initial optical density or X-ray absorption of the suspension is recorded. This corresponds to 100 % cumulative undersize. Before starting a test, the instrument is zeroed so