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Microbeam analysis — Methods of specimen preparation for analysis of general powders using WDS and EDS

Analyse par microfaisceaux — Méthodes de préparation des échantillons pour l'analyse des particules

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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Foreword

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This document was prepared by Technical Committee ISO/TC 202, *Microbeam analysis*.

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.

Introduction

Although there are many applications of electron probe microanalysis (EPMA) and scanning electron microscopy (SEM) for powder analysis, there are some difficulties, especially in the case of individual particle analysis, as follows:

(a) the prevention of agglomeration of particles during preparation of the specimen;

(b) the fixation of specimens, especially when there is a small amount of tiny particles, either for surface analysis or cross-section analysis;

(c) the cross-section preparation in the case of small particles with core-shell structures;

(d) the protection of particle surfaces from damage by electron beam irradiation in cases where the surfaces of particles are sensitive;

(e) the counteraction of charging of the specimen under electron radiation to prevent the powder from scattering or dispersing due to electrical repulsion;

(f) the interpretation of qualitative and/or quantitative analysis results when the X-ray generation volume is larger than that of the particles.

Even in the case of elemental compositional analysis of a powder, the specimen preparation can affect the results of analysis, because the roughness and/or void space within a particle aggregate or agglomerate can impact X-ray intensity.

To cope with these difficulties, the standardization of specimen preparation for particle analysis is very important.

Microbeam analysis — Methods of specimen preparation for analysis of general powders using WDS and EDS

1 Scope

This document specifies specimen preparation methods for the analysis of particles in powders using energy-dispersive spectrometers (EDS) or wavelength-dispersive spectrometers (WDS) installed on an EPMA or SEM. The preparation methods for powder particle analysis are classified by the analytical purpose and the particle size.

This document applies to inorganic particles larger than 100 nm and smaller than 100 µm in diameter.

It applies only to analysis of "general" powders, which means that it excludes procedures for special applications such as forensic or trace analysis.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

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/

4 Abbreviated terms

EDS	energy-dispersive X-ray spectroscopy/spectrometry
EPMA	electron probe microanalysis/electron probe microanalyzer
SEM	scanning electron microscopy/scanning electron microscope
WDS	wavelength-dispersive X-ray spectroscopy/spectrometry/spectrometer

5 Analytical purposes and methods of specimen preparation for particle analysis[1]

5.1 Methods of specimen preparation for particle analysis

The following methods of specimen preparation are widely used for particle analysis (see Figure 1). The specific procedure is indicated in 5.2.

This list is not comprehensive and does not preclude the use of other specimen preparation methods for particle analysis that can be more appropriate in some cases.

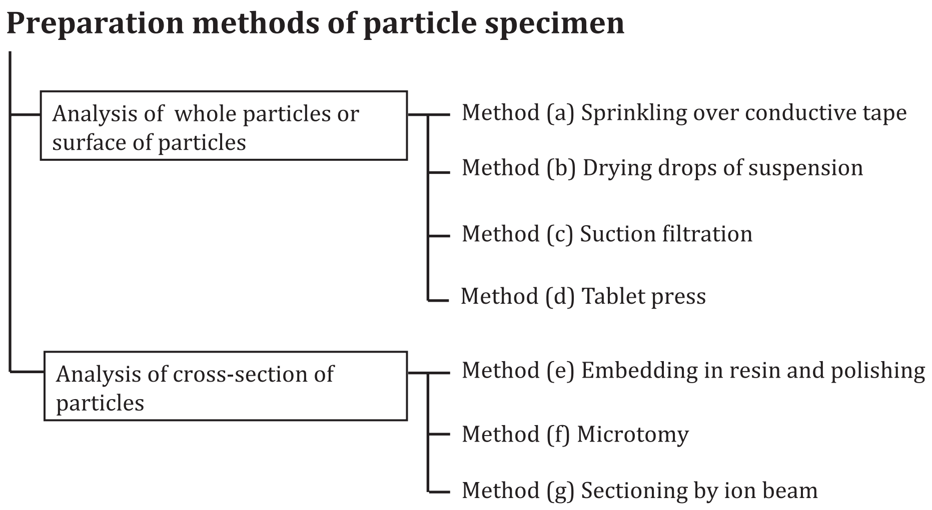

Figure 1 — Methods of specimen preparation for particle analysis

5.2 Description of preparation methods

5.2.1 Analysis of whole particles or surface of particles

For the analysis of whole particles or the surface of particles, methods (a), (b), (c) and (d) are adopted (see Figure 2).

— Method (a) Sprinkling over conductive tape:

  Place a piece of conductive tape on a conductive substrate, then sprinkle the specimen powder onto the conductive tape. (Before sprinkling the powder, it is better to put the substrate into a vacuum to remove any air beneath the conductive tape.) Remove any poorly-adhered material, for example by blowing away extra powder with an air duster or by turning the mount on its side and sharply tapping it.

— Method (b) Drying drops of suspension (for agglomerated powder):

  Suspend the powder in alcohol or water and drop the suspension liquid onto a metallic specimen holder. Next, dry the suspension liquid. After suspending the powder in the liquid, it is useful in many cases to remove agglomerations by centrifugation.

— Method (c) Suction filtration (for powder suspended in a liquid):

  Drop the liquid with floating powder onto the filter. After the suction filtration, dry the filter.

  When performing a filtration, it can be effective to use a filter funnel, fritted glass disk filter support or stainless steel mesh filter support. When using an ultrasonic bath, ensure that the ultrasonication has removed any agglomeration of particles in the suspension.

— Method (d) Tablet press (for a minute amount of powder):

  Place an extender on the lower die of a tablet press and make a dimple on the surface of the extender. Next, put the powder onto the dimple and press from both sides to make a tablet. Finally, remove the tablet from the die set.

  By sufficiently pressing and solidifying the powder, quantitative accuracy similar to that of solids can be obtained, even for powder. However, if powder pressing is insufficient then quantitative analysis accuracy is affected.

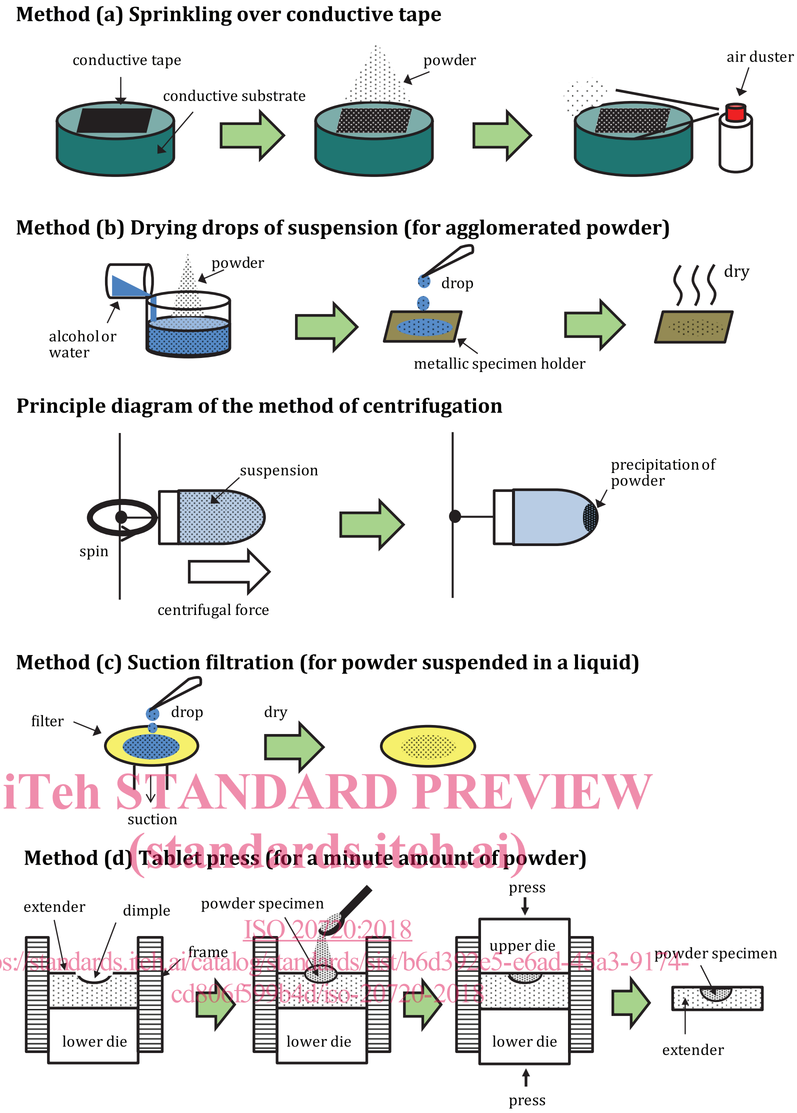

Figure 2 — Description of preparation methods (analysis of whole particles or surface of particles)

For method (d), silver flakes may be used when analyzing individual particles[2].

Place the silver flakes on the lower die of a tablet press. Next, sprinkle the specimen powder onto the silver flakes without making a dimple on the surface of the flakes. It is better to use tin flakes if the powder specimen is a sulfide, because sulfides react with silver. Press the mixture at a pressure of about 1,4 × 10^3 GPa and mildly anneal to densify. Keep the disk together for further processing. Finally, grind and polish the composite disk to reveal the polished analyte surface.

The surface conditions of tablets made using the press method differ depending on composition, particle size of the powder specimen and press pressure. The X-ray strength detected by EDS and WDS analysis is influenced by the condition of the specimen; therefore, it is important to choose an appropriate press pressure in press forming and to remove any voids in the tablet formed from the particles (see Annexes A and B).