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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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Foreword

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

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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.

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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.

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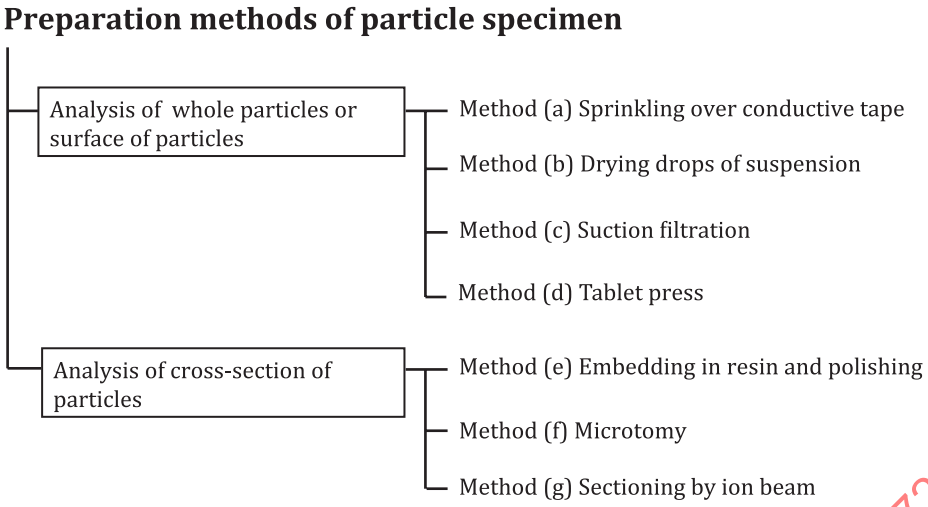

Figure 1 — Methods of specimen preparation for particle analysis

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.

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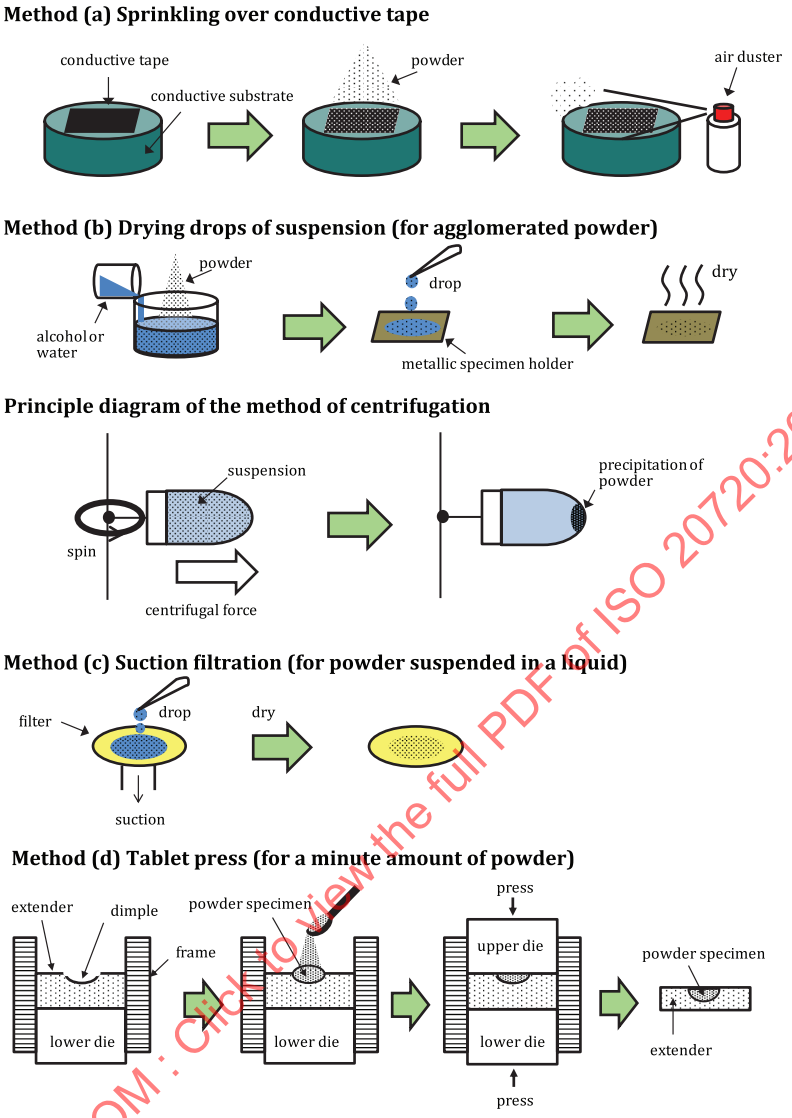

Figure 2 — Description of preparation methods (analysis of whole particles or surface of particles)

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 $1{,}4 \times 10^3$ 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).

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5.2.2 Analysis of cross-section of particles

For analysis of the cross-section of particles, methods (e), (f) and (g) are used (see Figure 3).

— Method (e) Embedding in resin and polishing:

Mix the specimen powder with resin and place it in a moulding frame. Then add resin to the mixture of resin and powder. After the resin has hardened, remove the mould and polish to expose a cross-section of powders.

When the powder is mixed with resin, first add a large amount of powder to a small amount of resin and mix it thoroughly so that the powder is uniformly dispersed in the resin.

For thermal-sensitive particles, a resin shall be chosen that does not cause the temperature to rise during hardening.

The method of embedding in resin and polishing is suitable for observing the cross-sectional structure of particles. Quantitative analysis is also possible if the particle size is large enough. The analysis area and depth of electron penetration can be estimated by referring to ISO 14594:2014, Annex A and Annex B, respectively.

— Method (f) Microtomy:

Mix powder with resin. After the resin has hardened, cut it using a microtome.

For method (e) and method (f), it can be helpful in some cases to use a conductive resin to impart conductivity to the specimen.

— Method (g) Sectioning by ion beam:

Make a pellet of powder and resin in the same manner as for method (e). After the resin has hardened, etch the surface of the pellet using an ion beam.

There are two methods of etching. The first is by using a focused ion beam (FIB). FIB preparation allows the sectioning to target individual particles. The second is sectioning using a broad argon ion beam, as described in Figure 3. This allows sectioning of many particles at once.

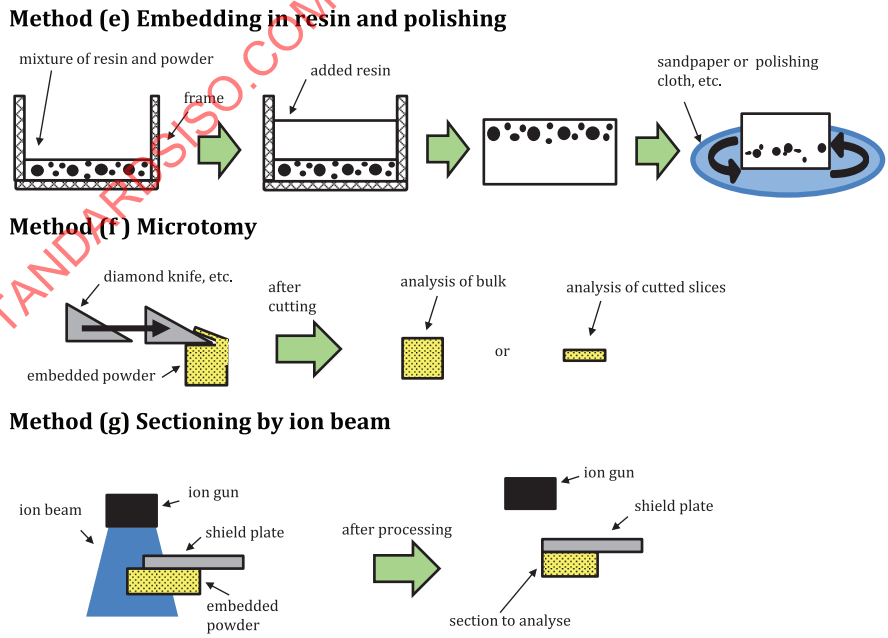

Figure 3 — Description of preparation methods (analysis of cross-section of particles)

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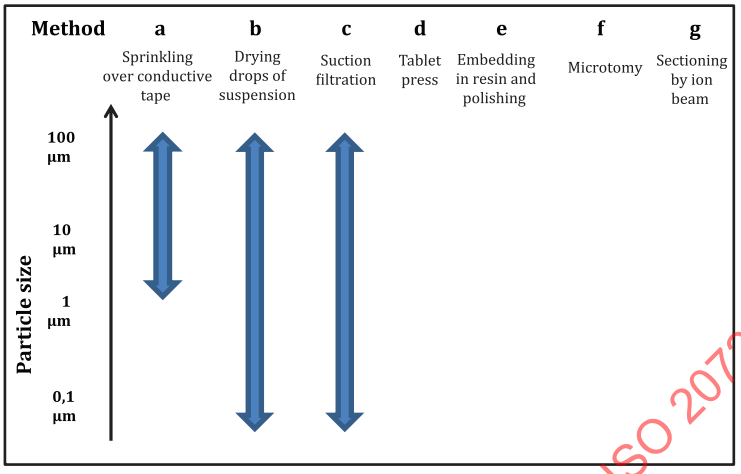
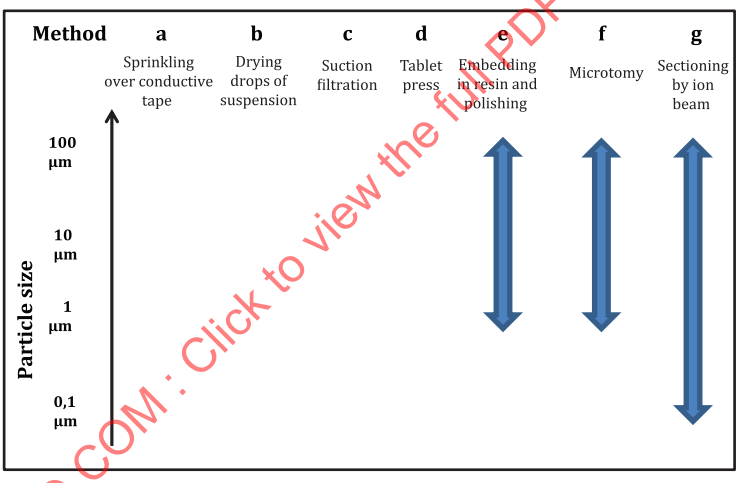
5.3 Choosing preparation methods

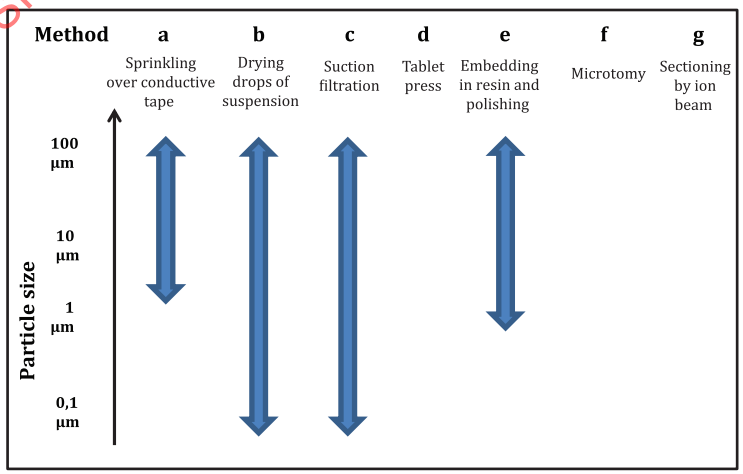
Figure 4, (a) to (d), should be used to choose an appropriate preparation method according to the analysis purpose and the particle size of the specimen.



(a) Analysis of surface of particles



(b) Analysis of cross-section of particles



(c) Analysis of agglomerated particles

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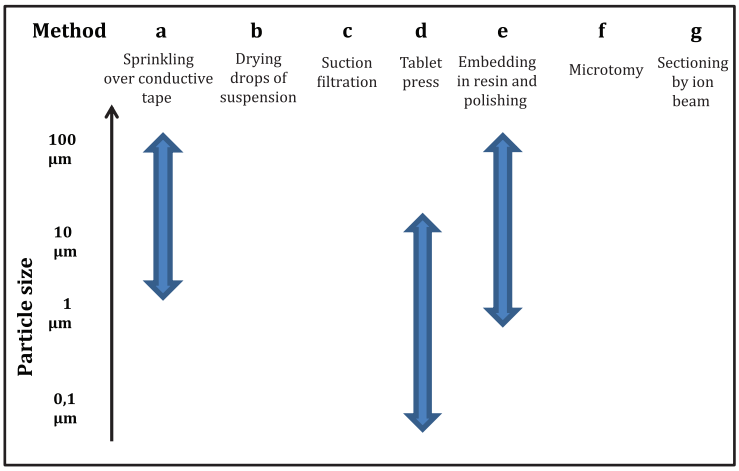

(d) Statistical analysis (e.g. type/size/distribution of particles)

Figure 4 — Diagrams for choosing preparation methods

6 Electric conductivity processing

For analysis of a non-conductivity specimen, an electroconductive metal or carbon is required to be coated on the surface of the specimen (see ISO 22489:2016, 4.2).

Vacuum deposition and ion plating are the two main methods used to secure conductivity. They are widely used in microbeam analysis and an appropriate method is selected according to the analysis purpose. Typical principles of conductivity processing methods and their features are illustrated in Figure 5.

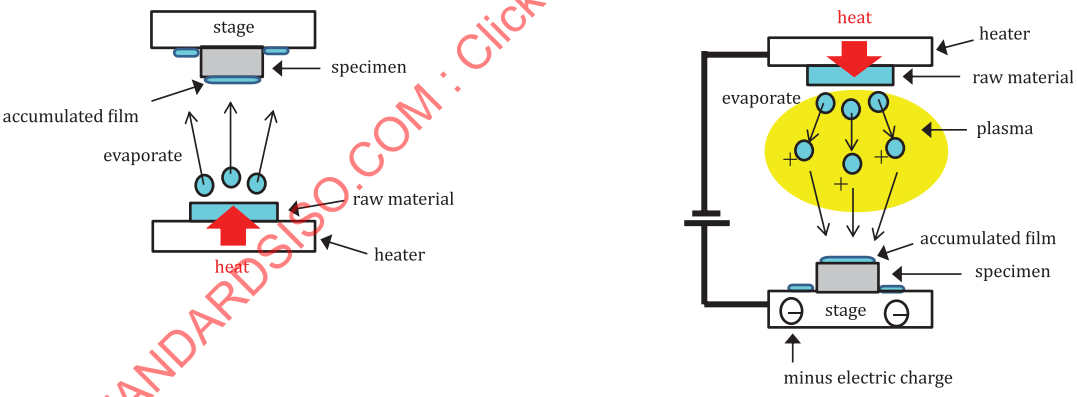

Figure 5 — Methods of (a) vacuum deposition and (b) ion plating

Particles sensitive to heat or energy transfer through an electron beam shall be protected by an additional layer. This layer shall have a good thermal conductivity, such as that provided by Au or Pt. In preparation method (g), a protective layer can be produced by platinum deposition in a dual-beam or cross-beam device using a gas injection system (GIS). To protect the structure of the surface of the particles, the deposition can be carried out using electron-beam-induced deposition.

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