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Metallic coatings — Measurement of coating thickness — Scanning electron microscope method

Revêtements métalliques — Mesurage de l'épaisseur de revêtement — Méthode au microscope électronique à balayage

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 9220 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*.

Annex A of this International Standard is for information only.

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Metallic coatings — Measurement of coating thickness — Scanning electron microscope method

1 Scope

This International Standard specifies a method for the measurement of the local thickness of metallic coatings by examination of cross-sections with a scanning electron microscope (SEM). It is destructive and has an uncertainty of less than 10 % or 0,1 µm, whichever is greater. It can be used for thicknesses up to several millimetres, but it is usually more practical to use a light microscope (ISO 1463) when applicable.

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 listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1463 : 1982, *Metallic and oxide coatings — Measurement of coating thickness — Microscopical method.*

ISO 2064 : 1980, *Metallic and other non-organic coatings — Definitions and conventions concerning the measurement of thickness.*

3 Definition

For the purposes of this International Standard, the following definition applies.

local thickness: The mean of the thickness measurements, of which a specified number is made within a reference area. (See ISO 2064.)

4 Principle

A test specimen is cut, ground, and polished from a cross-section of the coating for metallographic examination by a scanning electron microscope. The measurement is made on a conventional micrograph or on a photograph of the video waveform signal for a single scan across the coating.

5 Instrumentation

5.1 Scanning electron microscope (SEM)

The SEM shall have a resolution capability of 50 nm or better. Suitable instruments are available commercially.

5.2 SEM stage micrometer

A stage micrometer or graticule is required for calibration of the magnification of the SEM. The stage micrometer or graticule shall have an uncertainty of less than 5 % for the magnification employed. Suitable stage micrometers or graticules are available commercially.

6 Factors influencing the measurement results

The following factors may affect the accuracy of a measurement of coating thickness.

6.1 Surface roughness

If the coating or its substrate is rough relative to the coating thickness, one or both of the interfaces bounding the coating cross-section may be too irregular to permit accurate measurement of the average thickness in the field of view.

6.2 Taper of cross-section

If the plane of the cross-section is not perpendicular to the plane of the coating, the measured thickness will be greater than the true thickness. For example, an inclination of 10° to the perpendicular will contribute a 1,5 % error.

6.3 Specimen tilt

Any tilt of the specimen (plane of cross-section) with respect to the SEM beam may result in an inaccurate measurement.

NOTE — If the tilt of the test specimen is different from that used for calibration, inaccuracies may result.

6.4 Coating deformation

Detrimental deformation of the coating can be caused by excessive temperature or pressure during the mounting and preparation of cross-sections of soft coatings or coatings that melt at low temperatures, and by excessive abrasion of brittle materials during preparation of cross-sections.

6.5 Rounding of edges of the coating

If the edge of the coating cross-section is rounded, i.e. if the coating cross-section is not completely flat up to its edges, the observed thickness may differ from the true thickness. Edge rounding can be caused by improper mounting, grinding, polishing, or etching (see 6.6 and clause A.1).

6.6 Overplating

Overplating of the test specimen serves to protect the coating edges during preparation of cross-sections and thus to prevent an inaccurate measurement. Removal of the coating material during surface preparation for overplating can cause a low thickness measurement.

6.7 Etching

Optimum etching will produce a clearly defined and narrow dark line at the interface between the two metals. A wide or poorly defined line can result in an inaccurate meaurement.

6.8 Smearing

Polishing may leave smeared metal that obscures the true boundary between two metals and results in an inaccurate measurement. This may occur with soft metals like lead, indium, and gold. To help identify whether or not there is smearing, repeat the polishing, etching, and measurement several times. Any significant variation in readings is an indication of possible smearing.

6.9 Poor contrast

The visual contrast between metals in an SEM is poor when their atomic numbers are close together. For example, bright and semi-bright nickel layers may not be discriminable unless their common boundary can be brought out sufficiently by appropriate etching and SEM techniques. For some metal combinations, energy dispersive X-ray techniques (see A.3.5) or backscatter images (see A.3.6) can be helpful.

6.10 Magnification

For a given coating thickness, measurement errors tend to increase with decreasing magnification. If practical, the magnification should be chosen so that the field of view is between 1,5 and 3 times the coating thickness.

The magnification readout of an SEM often differs from the actual magnification by more than the 5 % often quoted and, for some instruments, the magnification has been found to vary by 25 % across the field. Magnification errors are minimized by appropriate use of an SEM stage micrometer.

6.11 Uniformity of magnification

Because the magnification may not be uniform over the entire field, errors can occur if both the calibration and the measurement are not made over the same portion of the field. These errors can be very significant.

6.12 Stability of magnification

6.12.1 The magnification of an SEM may drift with time. This effect is minimized by mounting the stage micrometer and test specimen side by side on the SEM stage so as to keep the transfer time short.

6.12.2 A change in magnification can occur when adjustments are made with the focusing and other SEM electronic controls; for example the scan rotation, operating voltage and contrast controls.

Such a change is prevented by not using the focus controls or other SEM electronic controls after photographing the stage micrometer scale except to focus using the x, y and z controls of the stage. Appropriate manipulation of the x, y and z controls will bring the specimen surface to the focal point of the SEM beam.

6.13 Stability of micrographs

Dimensional changes of micrographs can take place with time and with temperature and humidity changes. If the calibration micrograph of the stage micrometer scale and the micrograph of the test specimen are kept together and time is allowed for stabilization of the photographic paper, errors from this source will be minimized. The use of resin-coated paper is advised.

7 Preparation of cross-sections

Prepare the test specimen so that

a) the cross-section is perpendicular to the plane of the coating;

b) the surface is flat and the entire width of the coating image is simultaneously in focus at the magnification to be used for the measurement;

c) all material deformed by cutting or cross-sectioning is removed;

d) the boundaries of the coating cross-section are sharply defined by no more than contrasting appearance, or by a narrow, well-defined line;

e) if the video waveform signal is to be measured, the signal trace is flat except across the two boundaries of the coating.

NOTE — Further guidance is given in annex A.

8 Calibration of instruments

8.1 General

Before use, each instrument (5.1) shall be calibrated with an SEM stage micrometer or graticule (5.2) using a photograph taken under the same conditions as used for the sample measurement.

Appropriate attention shall be given to the factors listed in clause 6, to the procedures specified in clause 9, and to the uncertainty limits of clause 10. The stability of the calibration shall be checked at frequent intervals.

8.2 Photography

Photograph the image of the micrometer scale using a minimum signal-to-noise ratio of 2 to 1 and with sufficient image contrast for later measurement.

8.3 Measurement

8.3.1 Measure the perpendicular centre-to-centre distance between the lines in the photographed image to the nearest 0,1 mm. Use a diffraction plate reader or equivalent for this measurement.

8.3.2 Repeat the measurement at at least three different locations at least 3 mm apart on the photograph to determine the average spacing.

8.4 Calculation of magnification

Calculate the magnification of the photograph by dividing the average of the measurements between selected lines by the certified distance between the lines:

$$\gamma = \frac{l_m}{l_c} \times 1\,000$$

where

γ is the magnification;

l_m is the measured distance, in millimetres, on the photograph (average of measurements);

l_c is the certified distance, in micrometres.

9 Procedure

9.1 Each instrument (5.1) shall be operated in accordance with the manufacturer's instructions. Appropriate attention shall be given to the factors listed in clause 6 and to the uncertainty requirements of clause 10.

9.2 Make a micrograph of the test specimen under the same conditions and instrument settings as used for the calibration and make an appropriate measurement of the micrograph image. Carry out this step in accordance with 9.2.1 or 9.2.2.

9.2.1 Conventional micrograph

9.2.1.1 With the boundaries of the coatings clearly and sharply defined, make conventional micrographs of the SEM stage micrometer scale and of the test specimen.

9.2.1.2 Measure the micrographs to at least the nearest 0,1 mm using a diffraction plate reader or other optical device for making accurate linear measurements on film or paper. If this is not practical, the sample preparation may not have been suitable.

9.2.2 Video waveform signal

9.2.2.1 Photograph the video waveform signal for a signal scan across the coating cross-section and across the SEM stage micrometer scale.

9.2.2.2 To measure the coating, measure the horizontal distance between the inflection point of the vertical portions of the scan at the boundaries of the coatings. Make the measurements to the nearest 0,1 mm using a diffraction plate reader or equivalent device.

NOTE — Further guidance is given in annex A.

9.3 Calculate the thickness from the equation

$$d = \frac{l_m}{\gamma} \times 1\,000$$

where

d is the coating thickness, in micrometres;

l_m is the linear distance, in millimetres, on the micrograph;

γ is the magnification factor (see 8.4).

10 Measurement uncertainty

The instrument, its calibration, and its operation shall be such that the uncertainty of the coating thickness measurements is less than 10 % or 0,1 µm, whichever is greater (see A.3.7).

11 Expression of results

Express the results in micrometres to the nearest 0,01 µm, but with three digits if greater than 1 µm.

NOTE — This requirement is intended to minimize measurement uncertainty due to rounding of calculated values.

12 Test report

The test report shall contain at least the following information:

a) reference to this International Standard;

b) the measured value;

c) identification of the test specimen(s);

d) location of the measurements on the test specimen(s);

e) the magnification as measured before and after the test specimen measurements;

f) any unusual features of the measurements that may have affected the results;

g) date the measurements were made;

h) name of the individual responsible for the measurements;

i) type of measurements: conventional micrograph or video waveform signal.

Annex A
(informative)

General guidance on the preparation and measurement of cross-sections

A.0 Introduction

The preparation of specimens and measurements of coating thickness are greatly dependent on individual techniques and there is a variety of suitable techniques available. It is not reasonable to specify only one set of techniques, and it is impractical to include all the suitable techniques. The techniques described in this annex are intended for guidance only.

A.1 Mounting

To prevent rounding of the edges of the coating cross-section, the free surface of the coating should be supported so that there is no space between the coating and its support. This is usually achieved by overplating the coating with at least 10 µm of a metal with a hardness similar to that of the coating. The overplate should also give an electron signal different from that of the coating.

The surface of the mounting material has to be made electrically conducting to prevent a charge build-up.

If very soft materials are being prepared, abrasive particles may become embedded during grinding. This may be minimized by totally immersing abrasive papers in a lubricant during grinding or by using a copious flow of lubricant. If abrasive particles do become embedded, they may be removed by applying a short, light hand polish with metal polish after grinding and before diamond finishing or by one or more cycles of alternate etching and polishing.

A.2 Grinding and polishing

A.2.1 It is essential to keep the cross-section surface of the mount perpendicular to the coating. This is facilitated by incorporating additional pieces of a similar metal in the plastic mounting near the outer edges, by periodically changing the direction of grinding (rotating through 90°), and by keeping the grinding time and pressure to a minimum. If, before grinding, reference marks are inscribed on the sides of the mounts, any inclination from the horizontal is easily measured. Grind the mounted specimens on suitable abrasive paper, using an acceptable lubricant such as water or white spirit, and apply minimum pressure to avoid bevelling the surface. Initial grinding should employ 100 or 180 grade abrasive to reveal the true specimen profile and to remove any deformed metal. Subsequently, use grades 240, 320, 500 and 600 without exceeding grinding times of 30 to 40 s on each paper; alter the direction of grinding by 90° for each change of paper. Then polishing successively with 6 to 9 µm, 1 µm, and 0,5 µm diamond on microcloth is often recommended.

A.2.2 A convenient way to check for tapering of the cross-section is to mount a small diameter rod or wire with the specimen so that the perpendicular cross-section of the rod is parallel to that of the coating. If a taper is present, the cross-section of the rod will be elliptical.

A.2.3 If the video waveform signal scan technique is used, it is important that scratches be completely removed and that overpolishing does not selectively remove one of the metals more than the other so that the signal scan is distorted. With careful polishing it is unnecessary to use chemical etches.

A.3 Use of the scanning electron microscope

A.3.1 If the image of the cross-section, as revealed in a conventional micrograph, is to be measured, and if the boundaries of the coating cross-section are revealed solely by the photographed contrast between the two materials, the apparent width of the coating cross-section can vary depending on the contrast and brightness settings. The variation can be as great as 10 % without any change in instrument magnification. To minimize the resulting uncertainty, adjust the contrast and brightness so that the image shows surface detail of the materials on either side of each boundary.

A.3.2 Because the magnification of an SEM can change spontaneously with time and can change as a result of changing other instrument settings, it is advisable to calibrate the instrument immediately before or after measurement of the test specimen. For critical measurements, the average of calibration measurements made before and after measurement of the specimen should be used. This ensures that no change in the magnification has occurred and provides information on the precision of the calibration.

A.3.3 If the video waveform trace is measured, the measurement is made of the horizontal distance between the inflection points at the boundaries. The inflection point is halfway between the horizontal traces of the two materials. Because this horizontal distance is independent of the contrast and brightness settings and is precisely defined, some operators prefer to measure the video waveform trace for accurate measurements at higher magnifications.

A.3.4 For a video waveform trace, select a portion of the polished specimen that yields a flat, smooth signal.

A.3.5 Many SEMs are equipped with energy dispersive X-ray spectroscopy (EDS) which can be helpful in identifying the metal coating layers. At best the resolution of EDS is about 1 µm and is often poorer.

A.3.6 The use of backscatter images instead of secondary electron images can also be helpful in distinguishing metal layers with atomic numbers as close together as 1, and with a resolution of 0,1 µm.

A.3.7 A comprehensive investigation of measurement errors has not been reported. For a thin gold coating, one laboratory reported a measurement uncertainty of 0,039 µm for the certification of the SEM stage micrometer scale, 0,02 µm for the measurement of the calibration micrographs, and 0,02 µm for the measurement of the video waveform signal scan.

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