International Standard 1463

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Metallic and oxide coatings — Measurement of coating thickness — Microscopical method

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Foreword

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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.

International Standard ISO 1463 was developed by Technical Committee ISO/TC 107, *Metallic and other non-organic coatings*, and was circulated to the member bodies in November 1980.

It has been approved by the member bodies of the following countries :

Australia	Italy	Spain
Bulgaria	Japan	Sweden
Czechoslovakia	Netherlands	Switzerland
Egypt, Arab Rep. of	New Zealand	United Kingdom
France	Poland	USA
Germany, F. R.	Portugal	USSR
Hungary	Romania	Venezuela
India	South Africa, Rep. of	

No member body expressed disapproval of the document.

This second edition cancels and replaces the first edition (i.e. ISO 1463-1973).

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Metallic and oxide coatings — Measurement of coating thickness — Microscopical method

1 Scope and field of application

This International Standard specifies a method for the measurement of the local thickness of metallic coatings, oxide layers, and porcelain or vitreous enamel coatings, by the microscopical examination of cross-sections using an optical microscope.

Under good conditions, when using an optical microscope, the method is capable of giving an absolute measuring accuracy of 0,8 µm; this will determine the suitability of the method for measuring the thickness of thin coatings.

2 Reference

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

3 Definition

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

4 Principle

Cutting out a portion of the test specimen, mounting it, and preparing the mounted cross-section by suitable techniques of grinding, polishing, and etching. Measurement of the thickness of the coating cross-section by means of a calibrated scale.

NOTE — These techniques will be familiar to experienced metallographers, but some guidance is given in clause 5 and in annex A for less experienced operators.

5 Factors relating to the measuring accuracy

5.1 Surface roughness

If the coating or its substrate has a rough surface, one or both of the interfaces bounding the coating cross-section may be too irregular to permit accurate measurement. (See annex A, clause A.4.)

5.2 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.

5.3 Deformation of coating

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

5.4 Rounding of edge of 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 true thickness cannot be observed microscopically. Edge rounding can be caused by improper mounting, grinding, polishing or etching. It is usually minimized by overplating the test specimen before mounting. (See annex A, clause A.1.)

5.5 Overplating

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

5.6 Etching

Optimum etching will produce a clearly defined and narrow dark line at the interface of two metals. Excessive etching produces a poorly defined or wide line which may result in an erroneous measurement.

5.7 Smearing

Improper polishing may leave one metal smeared over the other metal so as to obscure the true boundary between the two metals. The apparent boundary may be poorly defined or very irregular instead of straight and well defined. To verify the absence of smearing, the coating thickness should be measured and the polishing, etching, and thickness measurement repeated. A significant change in apparent thickness indicates that smearing was probably present during one of the measurements.

5.8 Magnification

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

5.9 Calibration of stage micrometer

Any error in calibration of the stage micrometer will be reflected in the measurement of the specimen. Errors of several per cent are not unrealistic unless the scale has been calibrated or has been certified by a responsible supplier. A generally satisfactory means of calibration is to assume that the stated length of the full scale is correct, to measure each subdivision with a filar micrometer, and to calculate the length of each subdivision by simple proportion.

5.10 Calibration of micrometer eyepiece

A filar micrometer eyepiece generally provides the most satisfactory means of making the measurement of the specimen. The measurement will be no more accurate than the calibration of the eyepiece. As calibration is operator dependent, the eyepiece shall be calibrated by the person making the measurement.

Repeated calibrations of the micrometer eyepiece can be reasonably expected to have a spread of less than 1 %. The distance between the two lines of a stage micrometer used for the calibration shall be known to within 0,2 µm or 0,1 %, whichever is the greater. (The accuracy of some stage micrometers is certified by the manufacturer. Other stage micrometers have been found to be in error by 1 or 2 µm for a measurement distance of 2 mm and by 0,4 µm and more for measurement distances of 0,1 and 0,01 mm. If a stage micrometer is not certified for accuracy, it should be calibrated.)

Some image splitting micrometer eyepieces have a non-linearity which introduces an error of up to 1 % for short measurement distances.

5.11 Alignment

Errors can be introduced by backlash in the movement of the micrometer eyepiece. If the final motion during alignment of the hairline is always made in the same direction, this error will be eliminated.

5.12 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 with the measured boundaries centered about the optical axis.

5.13 Lens quality

Lack of sharpness of the image contributes to the uncertainty of the measurement. Poor quality lenses could preclude accurate measurements. Sometimes, image sharpness can be improved by using monochromatic light.

5.14 Orientation of eyepiece

The movement of the hairline of the eyepiece for alignment has to be perpendicular to the boundaries of the coating cross-section. For example, 10° misalignment will contribute a 1,5 % error.

5.15 Tube length

A change in tube length causes a change in magnification and, if this change occurs between the time of calibration and the time of measurement, the measurement will be in error. A change in tube length may occur when the eyepiece is repositioned within the tube, when the focus of the eyepiece tube is changed, and, for some microscopes, when the fine focus is adjusted.

6 Preparation of cross-sections

Prepare, mount, grind, polish, and etch the specimen so that :

a) the cross-section is perpendicular to 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.

NOTE — Further guidance is given in clause 5 and in annex A. Some typical etchants are described in annex B.

7 Measurement

7.1 Give appropriate attention to the factors listed in clause 5 and annex A.

7.2 Calibrate the microscope and its measuring device with a certified or calibrated stage micrometer.

7.3 Measure the width of the image of the coating cross-section at at least five points distributed along a length of the microsection.

8 Accuracy requirement

The microscope and associated equipment, its use, its calibration and the method of preparation of the cross-section shall be chosen so as to allow the coating thickness to be determined to within 1 µm or 10 %, whichever is the greater, of the actual coating thickness. Under good conditions, when using an optical microscope, the method is capable of giving an absolute measuring accuracy of 0,8 µm, and for thicknesses greater than 25 µm a reasonable error is of the order of 5 % or better.

9 Test report

The test report shall include the following information :

a) the location on the coated item at which the cross-section was made;

b) the measured thickness, in micrometres (millimetres if greater than 1 mm) at each point (7.3), and the length of section over which the measurements were distributed;

c) the local thickness, i.e. the arithmetic mean of the measured thicknesses.

Annex A

Guidance on the preparation and measurement of cross-sections

A.0 Introduction

The preparation of test specimens and measurement 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 suitable techniques. The techniques described in this annex are intended as guidance for metallographers not experienced in measurements of coating thickness.

A.1 Mounting

To prevent rounding of the edge 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 specimen with a coating at least 10 µm thick of a metal of similar hardness to the coating. For hard, brittle coatings (for example oxide or chromium coatings), tightly wrapping the specimen in soft aluminium foil before mounting has proved successful.

If the coating is soft, overplating with a metal which is softer will make polishing more difficult, because the softer metal tends to be polished away more rapidly.

Overplating of zinc or cadmium coatings with copper may cause difficulty because of the tendency, during subsequent etching, of dissolved copper to deposit on the coatings. It is better to overplate zinc with cadmium and vice versa.

A.2 Grinding and polishing

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 plastics 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 mount, any inclination from horizontal is easily measurable.

Grind the mounted test specimens on suitable abrasive paper, using an acceptable lubricant, such as water or white spirit, and apply minimum pressure to avoid bevelling of the surface. Initial grinding should employ 100 or 180 grade abrasive to reveal the true test 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 scratches by 90° for each change of paper. A final polish for 2 to 3 min on a rotating wheel charged with 4 to 8 µm diamond paste particles and lubrication with white spirit should suffice to remove scratches for final examination. If an especially high degree of surface finish is required, a further treatment, using diamond paste of approximately 1 µm particles, may be employed.

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.3 Etching

Etching is usually advisable in order to promote contrast between the metal layers, to remove traces of smeared metal and to develop a fine line at the boundary of the coating. Some typical etchants are given in annex B.

A.4 Measurement

The measuring device may be a filar micrometer or a micrometer eyepiece. The latter has a lower precision. An image splitting eyepiece is advantageous for thin coatings on rough substrate surfaces. Measurement of the image projected on a ground glass plate is usually less satisfactory because of the lack of sharpness of the image and poor legibility of the ruler when the projected image is visible.

The measuring device should be calibrated at least once before and once after a measurement, unless repeated experience indicates otherwise.

When making calibration and coating measurements, both should be made by the same operator, the stage micrometer and the coating should be centered in the field, and each measurement at a point should be made at least twice and averaged.

For critical and referee measurements, all steps for preparation of cross-sections and measurement of coating thickness, from grinding with 600 grade or coarser abrasive, up to and including the determination, should be performed at least twice. With good techniques and equipment, and smooth coating and substrate surfaces, repeatability within 2 % or 0,5 µm, whichever is the greater, is reasonable.

Some microscopes are subject to a spontaneous movement of the stage relative to the objective, possibly due to non-uniform thermal effects from the light source. Such a movement during the measurement can cause an erroneous measurement at moderate and high magnifications. This can be minimized by completing the measurement quickly and by measuring each interval twice, once from left to right and once from right to left.

Annex B

Some typical etchants for use at room temperature

WARNING — Precautions shall be taken in the preparation, use, handling and disposal of these etchants.

Etchant	Use and remarks
Etchant B.1 Nitric acid solution (ϱ = 1,42 g/ml) : 5 ml Ethanol, 95 % (V/V) solution : 95 ml **WARNING — This mixture can be explosively unstable, particularly if heated.**	For nickel or chromium coatings on steel. Etches steel. This etchant should be freshly prepared.
Etchant B.2 Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$) : 10 g Hydrochloric acid solution (ϱ = 1,16 g/ml) : 2 ml Ethanol, 95 % (V/V) solution : 98 ml	For gold, lead, silver, nickel and copper coatings on steel, copper, and copper alloys. Etches steel, copper, and copper alloys.
Etchant B.3 Nitric acid solution (ϱ = 1,42 g/ml) : 50 ml Glacial acetic acid : 50 ml	For determinations of thickness of individual layers of multi-layer coatings of nickel on steel and copper alloys; distinguishes each layer of nickel by identifying structures. Etches nickel; excessive attack on steel and copper alloys.
Etchant B.4 Ammonium persulphate : 10 g Ammonium hydroxide solution (ϱ = 0,88 g/ml) : 2 ml Distilled water : 90 ml	For tin and tin alloy coatings on copper and copper alloys. Etches copper and copper alloys. This etchant should be freshly prepared.
Etchant B.5 Nitric acid solution (ϱ = 1,42 g/ml) : 5 ml Hydrofluoric acid solution (ϱ = 1,14 g/ml) : 2 ml Distilled water : 93 ml	For nickel and copper coatings on aluminium and its alloys. Etches aluminium and its alloys.
Etchant B.6 Chromium(VI) oxide (CrO_3) : 20 g Sodium sulphate : 1,5 g Distilled water : 100 ml	For nickel and copper on zinc-based alloys. Also suitable for zinc and cadmium coatings on steel. Etches zinc, zinc-based alloys and cadmium.
Etchant B.7 Hydrofluoric acid solution (ϱ = 1,14 g/ml) : 2 ml Distilled water : 98 ml	For anodized aluminium alloys. Etches aluminium and its alloys.

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