SLOVENSKI STANDARD
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Kovinske in oksidne prevleke - Merjenje debeline prevleke - Mikroskopska metoda (ISO/DIS 1463:2020)

Metallic and oxide coatings - Measurement of coating thickness - Microscopical method (ISO/DIS 1463:2020)

Metall- und Oxidschichten - Schichtdickenmessung - Mikroskopisches Verfahren (ISO/DIS 1463:2020)

Revêtements métalliques et couches d'oxyde - Mesurage de l'épaisseur de revêtement - Méthode par coupe micrographique (ISO/DIS 1463:2020)

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

Revêtements métalliques et couches d'oxyde — Mesurage de l'épaisseur de revêtement — Méthode par coupe micrographique

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings.*

This third fourth edition cancels and replaces the third edition (ISO 1463:2003), which has been technically revised.

The main changes compared to the previous edition are as follows:

— digital image processing for light microscopes added;

— further hints and methods for the preparation of microsections added;

— some hazardous etching recipes removed from Annex C;

— editorial changes.

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.

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

1 Scope

This document 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.

WARNING — The use of this Standard can involve hazardous materials, operations and equipment. This Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of users of this standard to take appropriate measures to ensure the safety and health of personnel prior to application of the standard, and fulfil statutory and regulatory requirements for this purpose.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2064, *Metallic and other inorganic coatings — Definitions and conventions concerning the measurement of thickness*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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/

3.1
local thickness
the mean of the thickness measurements, of which a specified number is made within a reference area

[SOURCE: ISO 2064:1996, 3.4]

4 Principle

A portion of the test specimen is cut out and mounted. The mounted cross-section is prepared by suitable techniques of grinding, polishing and etching. The thickness of the coating cross-section is measured 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 measurement uncertainty

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 A.5).

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; e.g. an inclination of 10° to the perpendicular will contribute a 1,5 % uncertainty.

NOTE B.1 provides guidance on taper cross-section.

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 that melt at a low temperature, 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 A.2).

5.5 Overplating

Overplating of the test specimen protects the coating edges during preparation of cross-sections and thus prevents erroneous measurement. Removal of coating material during surface preparation for overplating can result in a low thickness measurement.

5.6 Etching

Optimum etching produces a clearly defined and narrow dark line at the interface of two metals. Excessive etching produces a poorly defined or wide line that can result in erroneous measurement.

5.7 Smearing

Improper polishing or overplating with a softer metal may cause smearing of one metal over the other metal, obscuring the boundary between the coating and the substrate. This problem may be alleviated by repeating the preparation of the cross section of the coated metal until repeatability of the thickness measurement (see A.3 and A.5) is obtained and also by overplating with a harder metal.

5.8 Magnification

For any given coating thickness, measurement uncertainty generally increases with decreasing magnification. The magnification should be chosen so that the field of view is between 1,5 × and 3 × the coating thickness.

5.9 Calibration of stage micrometer

Any uncertainty in calibration of the stage micrometer will be reflected in the measurement of the specimen. A suitable, traceable length standard shall be used.

5.10 Calibration of the microscope's length measuring device

5.10.1 Micrometer eyepiece

A filar micrometer eyepiece provides a 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.

Some image splitting micrometer eyepieces have a nonlinearity that introduces an uncertainty of up to 1 % for short measurement distances.

Uncertainties can be introduced by backlash in the movement of the micrometer eyepiece. To avoid this uncertainty, ensure that the final motion during alignment of the hairline is always made in the same direction.

5.10.2 Digital image processing

Microscopes with triocular tube, camera adapter with projecting lens and digital camera connected to a computer with a software for image capturing and processing are nowadays state-of-the-art. Similar to 5.10.1 the measurement will be no more accurate than the adjustment and calibration of the length measurement function (combination of hard- and software).

For adjustment digital images from the stage micrometer (in both directions parallel to the x- and y-axis of the image) are recorded for every combination of objective, eventual intermediate magnification changer, and resolution setting of the camera (full resolution and typical settings of pixel binning). The length in object space represented by a pixel of the digital image is calculated by measuring a known distance on the stage micrometer with the respective function of the software and is then saved in the software. Usually after such an adjustment the images are recorded "calibrated", i.e. with the µm/pixel factor assigned to the image, by selecting the objective, the eventual intermediate magnification changer, and the pixel setting of the camera in the software at the time of capturing the image.

The adjustment and/or calibration are usually stable for long time. Furthermore, they are not operator dependent as long as no changes are applied to the tube, an eventual intermediate magnification changer, the camera adapter, or the camera itself and as long as the same resolution of the camera (number of pixels in x and y direction) is used for adjustment and/or calibration and for measurement. Normally it is sufficient to record in regular time intervals images from the stage micrometer and measure known distances; when the deviation between the measured length and the certified length is less than a reasonably defined uncertainty limit for length measurements, which the laboratory wants to achieve, e.g. 1 %, the calibration is still valid and no re-adjustment is necessary.

5.11 Uniformity of magnification

Uncertainties can occur if the magnification is not uniform over the entire field of view. Thus ensure that both the calibration and the measurement are made over the same portion of the field of view with the measured boundaries centred about the optical axis.

5.12 Lens quality

As lack of sharpness of the image contributes to the uncertainty of the measurement, ensure that good quality lenses are used.

NOTE Sometimes, image sharpness can be improved by using monochromatic light.

5.13 Orientation of measuring lines

Ensure that the movement of the hairline of the eyepiece for alignment or the measuring line of a digital image processing software is perpendicular to the boundaries of the coating cross-section; e.g. 10° misalignment will contribute a 1,5 % uncertainties.

5.14 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 uncertainty. Take care to avoid a change in tube length, which can occur when the eyepiece is repositioned within the tube, when the focus of the eyepiece tube is changed, when the camera adapter is repositioned or 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 C.

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 on at least five points distributed along a length of the cross-section.

NOTE Guidance on the measurement of taper of cross-section and of tooth-constructed coatings is given in Annex B.

8 Measurement uncertainty

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. The method is capable of giving an absolute measurement uncertainty of 0,8 µm, and for thicknesses greater than 25 µm a reasonable measurement uncertainty is of the order of 5 % or better (see also B.3). However, with careful preparation of the specimen and the application of suitable instruments this method is capable of providing a measurement uncertainty of 0,4 µm under reproducible conditions.

