
INTERNATIONAL STANDARD



1463

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Metal and oxide coatings — Measurement of thickness by microscopical examination of cross-sections

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, International Standard ISO 1463 replaces ISO Recommendation R 1463-1970 drawn up by Technical Committee ISO/TC 107, *Metallic and other non-organic coatings*.

The Member Bodies of the following countries approved the Recommendation :

Australia	Iran	Spain
Chile	Israel	Sweden
Czechoslovakia	Italy	Switzerland
Egypt, Arab Rep. of	Netherlands	Thailand
Finland	New Zealand	Turkey
France	Norway	United Kingdom
Germany	Poland	U.S.A.
Hungary	Portugal	
India	South Africa, Rep. of	

No Member Body expressed disapproval of the Recommendation.

Metal and oxide coatings – Measurement of thickness by microscopical examination of cross-sections

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the measurement of thickness of metal coatings, oxide layers and porcelain or vitreous enamels by microscopical examination of a cross-section.

At best this method will yield an absolute measuring accuracy of $\pm 0,8 \mu\text{m}$; this will determine the suitability of the method for measuring the thickness of thin coatings.

2 SAMPLING

2.1 Specimens shall be taken from one or more points on the significant surface¹⁾. They shall be cut in such a way that the coating or oxide layer thickness is not altered.

2.2 To protect the edges of the specimen during preparation, soft coatings shall be overplated to a thickness of not less than $10 \mu\text{m}$ with a harder deposit showing good contrast when etched.

3 MOUNTING

3.1 The specimen shall be mounted to permit polishing of a section perpendicular to the surface where the coating or layer has to be measured²⁾.

3.2 The specimen shall be supported in a holder or mounting material in such a way that the temperature or pressure of the mounting operation does not significantly affect the thickness to be measured.

1) **significant surface** : The part of the surface which is essential to the appearance or serviceability of the article and which is to be covered or is covered by the coating.

When necessary, the significant surface shall be the subject of agreement, and shall be indicated on drawings, or by the provision of suitably marked samples.

2) A deviation of 10° from the perpendicular will produce an apparent thickness about 2 % higher than the actual thickness.

4 PREPARATION

4.1 The mounted specimen shall be ground and polished for microscopical examination using the lowest pressure compatible with the hardness of the coating.

4.2 If the direction of grinding can be controlled, it shall be from the harder to the softer material.

Grinding shall be continued until any irregularity produced during the cutting operation is completely removed.

Grinding strokes shall be angled at about 45° to the interface, the specimen being turned through 90° each time the abrasive is changed to a finer grade.

The ground surface shall then be polished on a low speed wheel with suitable media.

5 ETCHING

5.1 To obtain maximum contrast between coating(s) and basis metal, it is advisable to etch the polished section even if the contrast after polishing is apparently sufficient. Furthermore, etching removes any trace of soft metal which may have been smeared over the harder metal during previous operations.

In the case of aluminium oxide coatings, etching is not necessary but may be used.

5.2 Some typical etchants are listed in the Appendix.

6 MEASUREMENT

6.1 Measurement of the coating or layer thickness may be made by one of the following methods :

- a) observation of the specimen on a metallurgical microscope using a calibrated micrometer eyepiece;
- b) projection of the specimen image onto the screen of a metallurgical microscope at an accurately determined magnification. The actual thickness of the coating is obtained by measuring the projected coating thickness and dividing it by the magnification factor.

6.2 The number and distribution of the measurements along the micrographic section shall be decided in each individual case and stated in the report of the results.

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APPENDIX

Etchant	Usage and remarks
Nitric acid (HNO_3) ($d = 1,42$) – 5 parts by volume Ethyl alcohol ($\text{CH}_3\text{.CH}_2\text{OH}$) (95 %) – 95 parts by volume	For nickel or chromium deposits on steel. Etches the steel.
Ammonia solution ($\text{NH}_4 \text{OH}$) ($d = 0,90$) – 1 part by volume Hydrogen peroxide ($\text{H}_2 \text{O}_2$) (3 %) – 1 part by volume	For nickel deposits on copper and its alloys, including copper under-coats on ferrous metals and zinc alloys. Swab the specimen with a fresh solution. Etches the copper.
Chromic oxide (CrO_3) – 20 g Sodium sulphate (Na_2SO_4) – 1,5 g Distilled water – 100 ml	For zinc and cadmium deposits on steel, and nickel deposits on zinc alloys. Etches zinc and cadmium.
Ferric chloride ($\text{FeCl}_3\text{.6H}_2\text{O}$) – 10 g Hydrochloric acid (HCl) ($d = 1,16$ to $1,18$) – 2 ml Distilled water – 95 ml	For lead or copper deposits on steel. Etches the steel.
Nitric acid (HNO_3) ($d = 1,42$) – 1 part by volume Glacial acetic acid (CH_3COOH) – 1 part by volume	For multilayer deposits of nickel on steel and copper alloys; distinguishes each layer of nickel (dull, semi- bright or bright). Etches the nickel.
Hydrofluoric acid (HF) ($d = 1,14$) – 2 % by volume Distilled water – 98 % by volume	For anodized aluminium alloys. Etches aluminium and its alloys.

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