International Standard 4518

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Metallic coatings — Measurement of coating thickness — Profilometric method

Revêtements métalliques — Mesurage de l'épaisseur — Méthode profilométrique

First edition — 1980-07-15

UDC 669.058 : 531.717 **Ref. No. ISO 4518-1980 (E)**

Descriptors : metal coatings, dimensional measurement, thickness, measuring instruments, profile meters.

Price based on 4 pages

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 4518 was developed by Technical Committee ISO/TC 107, *Metallic and other non-organic coatings*, and was circulated to the member bodies in June 1978.

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

Czechoslovakia	Italy	Switzerland
France	Mexico	Turkey
Germany, F. R.	New Zealand	United Kingdom
Hungary	Poland	USA
India	Romania	USSR
Ireland	South Africa, Rep. of	
Israel	Sweden	

The member bodies of the following countries expressed disapproval of the document on technical grounds :

Japan
Netherlands

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Metallic coatings — Measurement of coating thickness — Profilometric method

1 Scope and field of application

1.1 This International Standard specifies a method for the measurement of metal coating thickness by first forming a step between the surface of the coating and the surface of its substrate and then measuring the step height using a profile recording instrument. It covers the instrumentation characteristics and the procedure appropriate to this specific application of profilometric methods.

1.2 The method is applicable to the measurement of thicknesses of metal coatings from 0,01 µm to 1 000 µm on flat surfaces and, if appropriate precautions are taken, on cylindrical surfaces. It is highly suitable for the measurement of minute thicknesses but, for thicknesses of less than 0,01 µm, surface flatness and surface smoothness are very critical and accordingly, the method is not recommended for use down to the lowest level of measurement usual for electronic stylus instruments. The method is suitable for measuring coating thicknesses when preparing coating thickness reference standards.

2 References

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

ISO 2177, *Metallic coatings — Measurement of coating thickness — Coulometric method by anodic dissolution.*

3 Principle

Formation of a step either by dissolving part of the coating (acceptance testing) or by masking a portion of the substrate prior to coating (production inspection). Measurement of the height of the step using a profile recording instrument.

4 Instrumentation : Operational parameters and measurement characteristics

4.1 Types of profile recording instruments

Either of two types may be used :

a) electronic stylus instruments, known as surface analysers and surface profile recorders, generally used to measure surface roughness but which, for the purposes of this International Standard, are used to record the profile of a step;

b) electronic inductive comparators equipped with styli and capable of recording the profile of a step.

Electronic stylus instruments may have a greater utility, being suitable for roughness measurements, while electronic inductive comparators may be simpler in construction. The two types of instrument generally cover different ranges of coating thickness : 0,005 to 250 µm for electronic stylus instruments, and 1 to 1 000 µm for electronic inductive comparators.

4.2 Electronic stylus instruments

4.2.1 These instruments are used to record the profile of a surface and have the following components.

4.2.1.1 A pick-up with a conical or pyramidal stylus having an included angle of 1,57 rad (90°) and a nominal tip radius, in the direction of the traverse, of 2, 5, 10 or 50 µm. The force of contact on the test surface shall not exceed the appropriate value given in the table.

Table — Force on stylus

Nominal value of stylus tip radius, µm	2	5	10	50**
Maximum static force at the mean level of the stylus, mN*	0,7	4	16	10**

* 1 mN ≈ 0,1 gf

** Values useful for low-hardness metals such as tin and lead.

4.2.1.2 A traverse unit that moves the pick-up relative to a datum skid or, in those cases where the skid may result in damage to the surface or introduce distortion of the step to be measured, a datum surface having nominal form of the profile.

4.2.1.3 An amplifying unit giving nominal values of the vertical (V_v) magnifications of the profile selected from the following series :

100 — 200 — 500 — 1 000 — 2 000 — 5 000 — 10 000 — 20 000 — 50 000 — 100 000 — 200 000 — 500 000 — 1 000 000.

4.2.1.4 A recording instrument that plots the amplified variations of the profile which, when operated in conjunction with the traverse unit, permits nominal values of the horizontal (V_h) magnifications of the profile selected from the following series :

10 — 20 — 50 — 100 — 200 — 500 — 1 000 — 2 000 — 5 000.

4.2.2 Profile recording instruments will furnish the following measuring characteristics :

- traverse length : 1 to 100 mm
- range of thickness measurement : 0,005 to 250 µm
- resolution (dependent on the range of measurement) : 0,000 5 to 1 µm.

4.3 Electronic inductive comparators

4.3.1 The design of electronic inductive comparators is much like that of the electronic stylus instruments (4.2), the principal difference being that the large-radius stylus does not plot the microprofile of the surface.

4.3.2 Typical examples of the measuring characteristics and the operational parameters of a comparator instrument are as follows :

An electronic inductive comparator with a linearity of not less than 0,5 %, a table providing a rectilinear motion to the surface to be traversed, and suitable amplifiers, and using the following operational parameters :

- radius of stylus : 250 µm
- maximum magnification : X 50 000
- static stylus force : 0,12 N

will furnish the following measurement characteristics :

- traverse length : 100 mm
- range of thickness measurement : 1 to 1 000 µm
- resolution (dependent on the range of measurement) : 0,02 to 20 µm.

5 Factors relating to accuracy

5.1 Profile record

Because the thickness measurement is made from the recorded profile, errors will arise if the recording does not give a faithful reproduction of the step at a suitable magnification. Inaccurate recordings may reflect the quality or improper adjustment of the recording instrument.

5.2 Vertical magnification

If the vertical magnification is too low, measurement precision will be poor. It should be set to take maximum advantage of the chart width.

5.3 Graphical measurements

If the test surface is not parallel to the reference (datum) surface, the recording of the horizontal surface is sloped with respect to the chart grid; the vertical portion of the step is also sloped but it may still be vertical on the chart grid, depending on the vertical and horizontal magnifications, on the radius of the stylus and finally on the height of the step (i.e. thickness). When the profile is sloped, a common error is to measure the perpendicular distance between the mean lines of the profile without correcting for the differences between the horizontal and vertical magnifications.

To avoid these errors or additional mathematical calculations, the datum and test surfaces shall be parallel. This may be accomplished by the use of an appropriate jig or fitting.

5.4 Applied force

If the force on the stylus is too great, the stylus produces a scratch or deformation which may introduce a measurement error. The force should be kept to a minimum and generally should not exceed the appropriate values given in the table in 4.2.1.1.

5.5 Stylus diameter and surface roughness

If a small-diameter stylus is used on a rough surface, the step height may be difficult to measure accurately because of poor definition of the extremes of the recorded step. A large-diameter stylus minimizes this difficulty.

If the substrate and coating surfaces are of different roughnesses, the recorded step profile may be misleading to the extent that the stylus rides the high spots more on one surface than the other because of different peak to peak spacings. A small-diameter stylus tends to reduce this error.

A small-diameter stylus equipped with an electronic filter to smooth out the profile can be advantageous, but may round the corners of the step profile.

In principle, the roughnesss of a recorded substrate (peak to valley height of the surface profile) should not exceed 10 % of the step height.

5.6 Vibrations

Vibrations may cause irregularities or noise in the recorded profile, making accurate measurement difficult. This effect should be minimized by insulating the equipment from vibrations. In principle, the peak to valley height should not exceed 10 % of the step height.

5.7 Surface curvature

Surface curvature can interfere with accurate measurement of

the recorded step profile. The measurement should be carried out on as flat a surface as practicable. If it has to be carried out on a curved surface, the traverse of the stylus should be in the direction of least curvature, for example parallel to the axis of a cylinder. (The step should be parallel to the direction of maximum curvature.)

5.8 Cleanliness

Any foreign matter such as dirt, grease, and corrosion products may lead to erroneous measurements. The surfaces to be measured should be cleaned, and the laboratory air should be reasonably free of dust and dirt.

5.9 Temperature

Temperature variations can affect the measurement. Therefore, the temperature should be uniform and reasonably stable.

5.10 Step configuration

A poorly defined step (for example excessive rounding of edges) can make an accurate measurement difficult by obscuring the levels of the upper and lower portions of the steps. The steps should be reasonably well defined.

5.11 Datum reference

The stylus holder rides on a datum skid or on a datum surface, and the vertical motion of the stylus relative to the datum skid or surface is recorded. The datum skid is a rounded surface which rides over the surface of the test specimen, and the datum surface is part of the equipment independent of the test specimen.

The faithfulness of a recording will depend on the quality (smoothness and straightness) of the datum reference.

5.12 Calibration

The thickness measurement will be not better than the uncertainty of the instrument calibration and that of the step height standard used to calibrate the instrument. The calibration can change, and the frequency with which calibration is required has to be learned by experience. Though the instrument may be carefully calibrated, an error of 2 % can exist because of nonlinearity of the instrument response. To minimize this error, the instrument can be calibrated at two points closely bracketing the step height to be measured.

6 Calibration

6.1 Calibrate the instrument in accordance with the manufacturer's instructions, paying appropriate attention to the factors listed in clause 5.

6.2 The step height of the standards used to calibrate the instrument should be known with an uncertainty of less than 5 %. However, for steps of less than 0,1 µm, the uncertainty may be considerably greater than 5 %.

6.3 Repeat the calibration at regular intervals (see 5.12), and whenever a calibration change is suspected.

7 Measuring procedure

7.1 Preparation of step

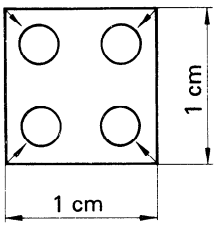
7.1.1 Remove a portion of the coating without any attack on its substrate. The top of the step shall not be marred or attacked in any way, and the bottom of the step shall be free of any traces of the coating. Suitable methods of producing steps are given in 7.1.2, 7.1.3 and 7.1.4.

7.1.2 Using a suitable material, mask all of the coating except the area to be dissolved. Dissolve the exposed coating in a suitable reagent which will not attack the substrate, and then remove all traces of the masking material.

7.1.3 Dissolve a small area of the coating with an electrolytic cell of the same type as those used for the coulometric measurement of thickness by anodic dissolution according to ISO 2177.

A single traverse of the stylus across the diameter of the circle produced by the small cell may provide a profile of two steps.

In order to be consistent with the definition in ISO 2064 of minimum thickness as measured over a reference area of about 1 cm^2, it is recommended to remove four small circular areas of coating within a 1 cm square and to record the step profile and to measure the step height nearest each of the four corners of the square.



7.1.4 In some cases, the step can be formed by masking a portion of the substrate prior to coating. The masked area shall be sufficiently small (diameter of the order of 1 or 2 mm) so that edge build-up will not interfere with the measurement.

7.2 Recording of profiles

Record the profile according to the instrument manufacturer's instructions, the operational parameters having been determined beforehand in accordance with the measurement characteristics sought and with clause 4. Particular attention shall be paid to the factors in clause 5 relating to measurement accuracy.

7.3 Measurement of thickness

Draw a mean line through each recording of the upper and lower levels of the step and extend them so that the two mean

lines overlap. Assess the step height from the two mean lines at the midpoint of the step.

When making this measurement, edge effects should be avoided and allowance should be made for variations in thickness of the film along the scanned profile.

7.4 Measurement accuracy

The instrument, its calibration and its operation shall be such that coating thickness can be determined to within 10 % or ± 0,005 µm, whichever is the greater.

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