
INTERNATIONAL STANDARD



3868

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Metallic and other non-organic coatings — Measurement of coating thicknesses — Fizeau multiple-beam interferometry method

Revêtements métalliques et autres revêtements non organiques — Mesurage de l'épaisseur — Méthode basée sur le principe de Fizeau d'interférométrie à faisceaux multiples

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Descriptors : coatings, metal coatings, dimensional measurement, thickness, optical measurement, interferometers.

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.

International Standard ISO 3868 was drawn up by Technical Committee ISO/TC 107, *Metallic and other non-organic coatings*, and was circulated to the Member Bodies in July 1975.

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It has been approved by the Member Bodies of the following countries:

| | | |
|----------------|-------------|-----------------------|
| Bulgaria | Italy | South Africa, Rep. of |
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| France | Mexico | Turkey |
| Germany | New Zealand | U.S.A. |
| Hungary | Poland | U.S.S.R. |
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The Member Body of the following country expressed disapproval of the document :

United Kingdom

Metallic and other non-organic coatings — Measurement of coating thicknesses — Fizeau multiple-beam interferometry method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the measurement of the thickness of thin, highly reflective coatings (up to $2\ \mu\text{m}$) by the use of Fizeau multiple-beam interferometry.

The method described cannot be applied to vitreous enamel coatings.

2 PRINCIPLE

By completely dissolving a small area of the coating without attack of its substrate (or by masking before plating), a step is formed from the surface of the coating to that of its substrate. The height of this step is measured with a multiple-beam interferometer.

A monochromatic light beam is reflected back and forth between the specimen surface and a superimposed transparent mirror serving as a planar reference plate, so as to produce a pattern of interference fringes observed through a low-power microscope. The reference plate is tilted slightly with respect to the surface being inspected, so that the fringe pattern is a series of parallel lines. A step in the specimen surface causes a shift in the fringe pattern. A shift of one full fringe spacing is equivalent to a vertical displacement of $1/2$ of the wavelength of the monochromatic light. The whole and fractional number of fringe spacings occupied by the fringe shift is determined with an eyepiece micrometer.

3 DEFINITIONS

3.1 filar micrometer eyepiece : A device for observing and measuring an image. It includes an adjustable lens, a hairline that is moved with a graduated knob, and a pattern of lines (graticule lines) across the field of view.

3.2 filar units : The graduations on the micrometer control which are proportional to the absolute unit of length.

3.3 Fizeau plate : An optically flat, smooth surface with high reflectivity and low absorption.

3.4 fringe lines : The dark lines caused by interference of light waves.

3.5 hairlines : The part of the filar eyepiece moved by means of the graduated knob to measure fringe line-spacing and offset.

3.6 offset : The displacement of a fringe line which occurs when it encounters a vertical variation on the surface of a specimen.

3.7 spacing : The distance between fringe lines.

4 EQUIPMENT

The instrument employs :

- a) a beam of monochromatic light;
- b) optics to direct the light through a specially coated Fizeau plate which comes into contact with the specimen at a slight angle and forms an air wedge. An interference fringe pattern is produced in the air wedge and viewed through a microscope equipped with a filar micrometer eyepiece. The spacing and shape of the fringe lines can be interpreted to determine an extremely accurate contour map of the specimen surface.

5 FACTORS AFFECTING THE MEASURING ACCURACY

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

5.1 Reflective overcoat

In order to obtain dark, narrow fringe lines necessary to achieve an accurate measurement, and in order to avoid errors due to different phase shifts, when light reflects over different materials, the test specimen shall be coated with a highly reflective material such as aluminium or silver. If the surfaces at the step are highly reflective and the errors due to phase shift are known and corrected for, then the reflective layer can be avoided.

5.2 Step form

For specimens with a coating thickness of less than $0,3 \mu\text{m}$ special fabrication is not normally necessary.

If the step being measured is abrupt, so that it is not possible to follow the fringes across the step, it will not be possible to observe how many full fringe intervals of displacement have occurred. This may be determined by an

independent estimate of thickness on the basis of prior knowledge, or on measurements by other techniques such as profilometry, white-light interferometry, etc.

By the appropriate method the stem can often be made less abrupt so that each fringe can be followed across each step. The optimum angle is normally in the range 95 to 100° .

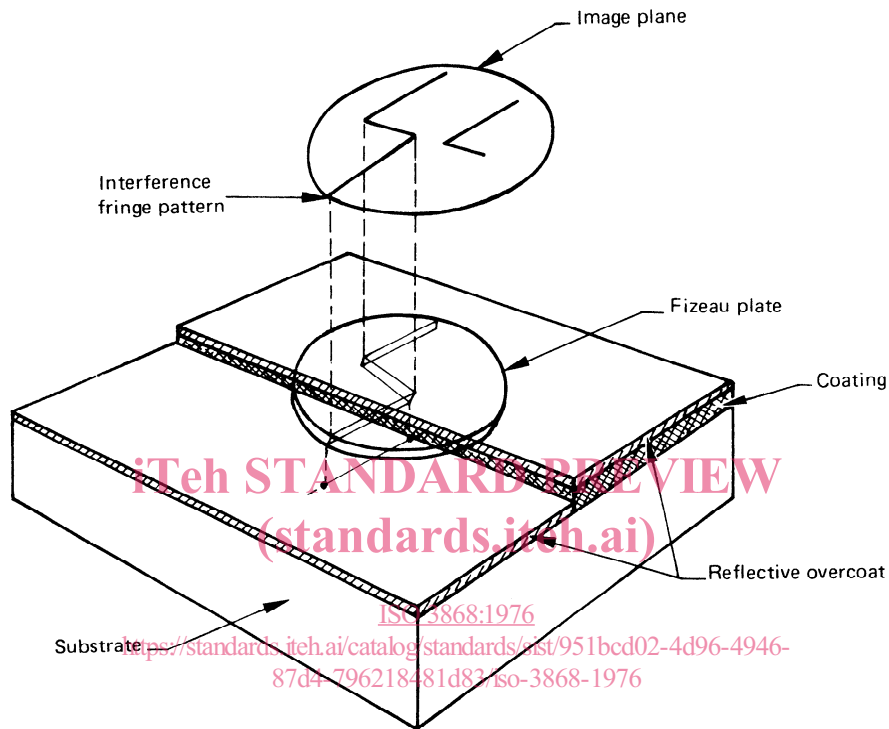


FIGURE 1

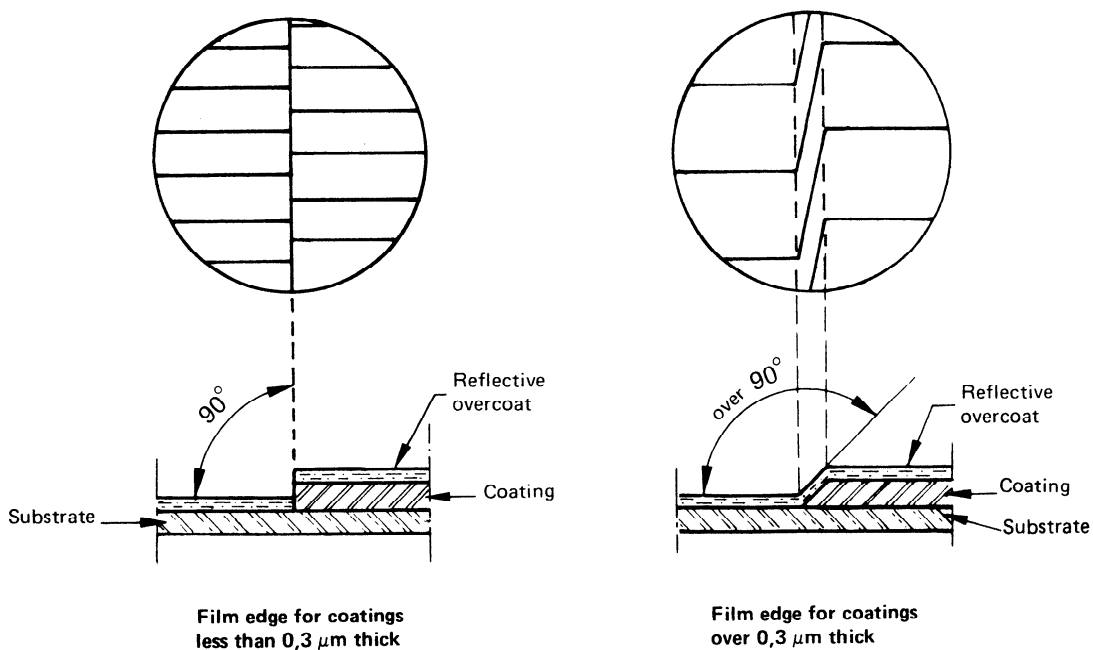


FIGURE 2

5.3 Reading accuracy

Possible backlash errors may be eliminated by always making the final adjustment of the micrometer in the same direction.

5.4 Surface flatness

A very flat surface is required, especially for measuring thicknesses between 0,001 and 0,01 μm , in order to produce lines of good definition when expanded. If the surface is not flat, the fringes may be curved, making accurate measurement more difficult. The loss of accuracy will increase as the radius of curvature decreases.

5.5 Step definition

In order to make good thickness measurements, it is necessary to have a good step definition. If the step is poorly defined the fringe lines are not straight and it is possible to make errors in the superposition of the hairline on the fringe line. Also it may be difficult or impossible to follow the fringes across the step. Poor definition may result from the step preparation method.

(If the coating whose thickness is to be measured has been obtained by electroplating, and if masking is done before deposition, there is likely to be a ridge or mound at the edge of the step because of local high current density along the edge. This will show up as a curve in the fringe pattern at the edge. This curve should be ignored or avoided.)

5.6 Roughness

Surface roughness such that the fringe lines become irregular causes uncertainty in measurement of coating thickness by making the fringes less sharp, very diffuse, and poorly defined.

5.7 Cleanliness

To achieve the best results it is necessary that the surface has no residues from manufacturing, fingerprints, oil, etc. Specimen areas having visible defects shall be avoided in making measurements.

5.8 Preparation of step

If the step is formed by masking and dissolution of the coating substrate or incomplete dissolution of the coating will lead to an erroneous measurement.

6 CALIBRATION

The measurement obtained by multiple-beam interferometry is absolute and calibration is not required.

7 PROCEDURE

7.1 Step preparation

7.1.1 Masking before plating

7.1.1.1 Mask a portion of the surface before plating.

7.1.1.2 Plate the unmasked portion of the surface.

7.1.1.3 Completely remove the masking material.

NOTE — The mask area should be as small as possible to minimize edge build-up.

7.1.2 Masking after plating

7.1.2.1 Mask all of the coating surface except that part to be dissolved.

7.1.2.2 Dissolve a small area of the coating.

7.1.2.3 Remove the masking material.

NOTES

1 The preparation of the step shall be such that the top of the step is not marred or attacked in any way, the bottom of the step is free of all traces of the coating, and the coating is removed without any attack whatsoever of its substrate.

2 The step shall slope so that the fringe displacement can be followed across the step. This requirement may be ignored if the step height or coating thickness is already known accurately enough to determine how many fringe spacings are covered by the fringe displacement.

7.2 Measuring procedure

To obtain a fringe-line pattern the following procedure shall be observed :

7.2.1 Adjust the object focus and place the step in the field of the microscope.

7.2.2 Adjust the angle between the Fizeau plate and the specimen to obtain a fringe-line pattern. Adjust the angular position of the specimen so that the fringes are perpendicular to the step.

7.2.3 Adjust the brightness, field size and eyepiece focus to obtain optimum definition of the fringes.

7.2.4 Adjust the focus of the object again for maximum clarity.

7.2.5 Measure the displacement of a fringe across the step and the spacing between fringes with a filar micrometer.

The coating thickness is given by

$$d = \Delta N \frac{\lambda}{2}$$

where

ΔN is the number of fringes or fraction traversing the step;

λ is the incident light wavelength.

In practice when making measurements with the filar eye-piece the fringe line offset method is used (see figure 3).

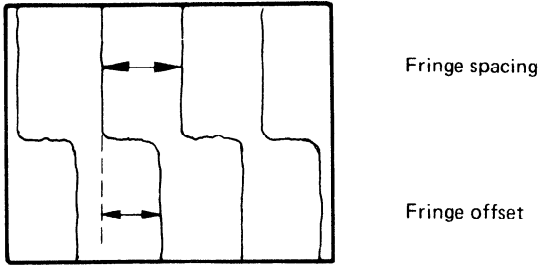


FIGURE 3

The coating thickness is calculated, with the fringe spacing and offset in filar units, in the following way :

$$\frac{\text{fringe offset (filar units)}}{\text{fringe spacing (filar units)}} \times \frac{\lambda}{2} = \text{height of surface variation}$$

For the measurement of thin coatings on smooth substrates an improvement can be made by using the fringe-line width method. This procedure is particularly useful for measuring surface variations between 0,002 and 0,01 μm but this technique also has general application. A very smooth specimen is necessary to obtain well-defined lines in order to retain good definition when expanded as shown in figures 4 and 5.

The procedure is as follows :

- 1) adjust the Fizeau plate tilt to spread the fringe line pattern until only two lines are in view (figure 4);

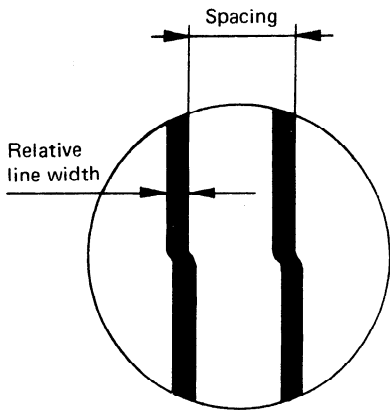


FIGURE 4

- 2) calculate the actual line width :

$$\frac{\text{relative line width (filar units)}}{\text{line spacing (filar units)}} \times \frac{\lambda}{2} = \text{actual line width}$$

- 3) adjust the Fizeau plate tilt to spread the fringe line pattern until only one broad fringe is in view (figure 5);

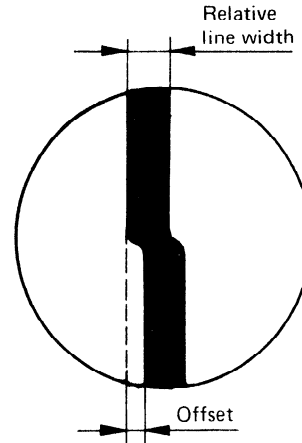


FIGURE 5

- 4) calculate the coating thickness :

$$\frac{\text{line offset (filar units)}}{\text{relative line width (filar units)}} \times \text{actual line width}^* = \text{film thickness}$$

The line width with a very smooth surface is about 0,006 μm. Accuracy is 0,003 μm, i.e. ± 50 %. Thus a film thickness of 0,002 μm can be measured with an accuracy of ± 0,001 μm.

The fringe-line width method is dependent on the fringe lines being of uniform width. The uniformity of line width should be verified experimentally or with the instrument manufacturer.

8 MEASURING PRECISION

The multiple-beam interferometer provides an absolute measure of the microscopic vertical surface variation ranging from 0,002 to 2 μm. Precision is normally ± 0,001 μm in the range 0,002 to 0,01 μm using the "fringe-line width method", while it is normally ± 0,003 μm in the range 0,01 to 2 μm.

* as calculated in step 2.

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