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Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of thickness of ceramic films by contact-probe profilometer

Céramiques techniques — Détermination de l'épaisseur des films céramiques avec un profilomètre à contact

Reference number
ISO 18452:2005(E)

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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.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 18452 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of thickness of ceramic films by contact-probe profilometer

1 Scope

This International Standard specifies a method for the determination of the film thickness of a fine ceramic film and ceramic coatings by a contact-probe profilometer. The method is suitable for film thicknesses in the range of 10 nm to 10 000 nm.

NOTE The method requires a distinct and clearly formed boundary between coated and uncoated parts of the substrate.

2 Normative references

The following referenced documents are indispensable for the application 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 3274, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Nominal characteristics of contact (stylus) instruments*

3 Terms and definitions

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

3.1

fine ceramic film

coating consisting of a fine ceramic material which thinly covers the substrate surface

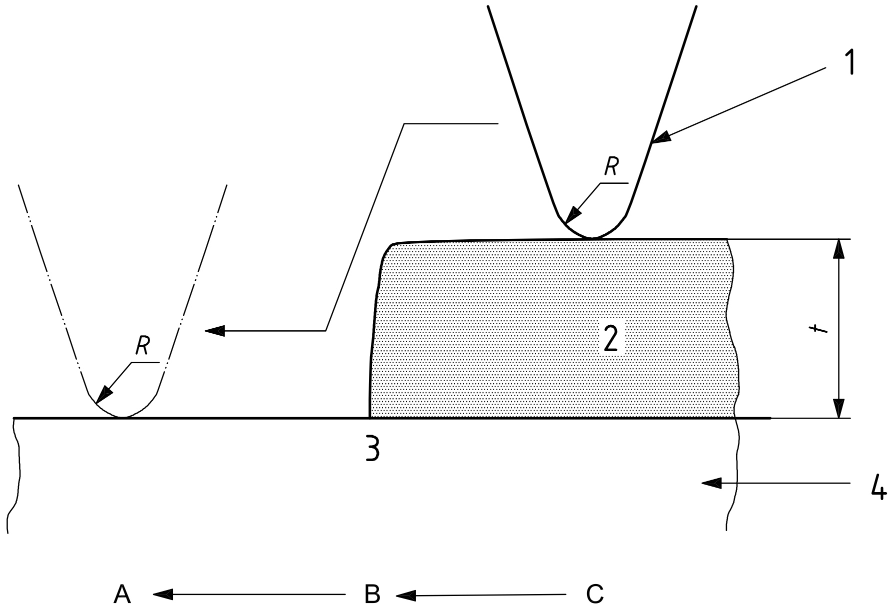
EXAMPLE Typical materials are oxides, carbides, nitrides, etc., deposited by methods such as vacuum evaporating, sputtering, chemical vapour deposition, etc.

4 Principle of measurement

This International Standard concerns the measurement of the film thickness of fine ceramic coatings on a substrate using a contact-probe profilometer. The film thickness shall be calculated from the profile which is obtained by scanning the contact probe in the direction C $\rightarrow$ B $\rightarrow$ A, as shown in Figure 1. The profile is in proportion to the difference in height between the parts covered and not covered with the fine ceramic film.

5 Test environment

The test shall be carried out in an environment free from mechanical vibrations that may affect the measurement.



Key

1 contact probe

2 film

3 level difference

4 substrate

t film thickness

Figure 1 — Principle of measurement using the contact-probe profilometer

6 Apparatus

6.1 Contact-probe profilometer

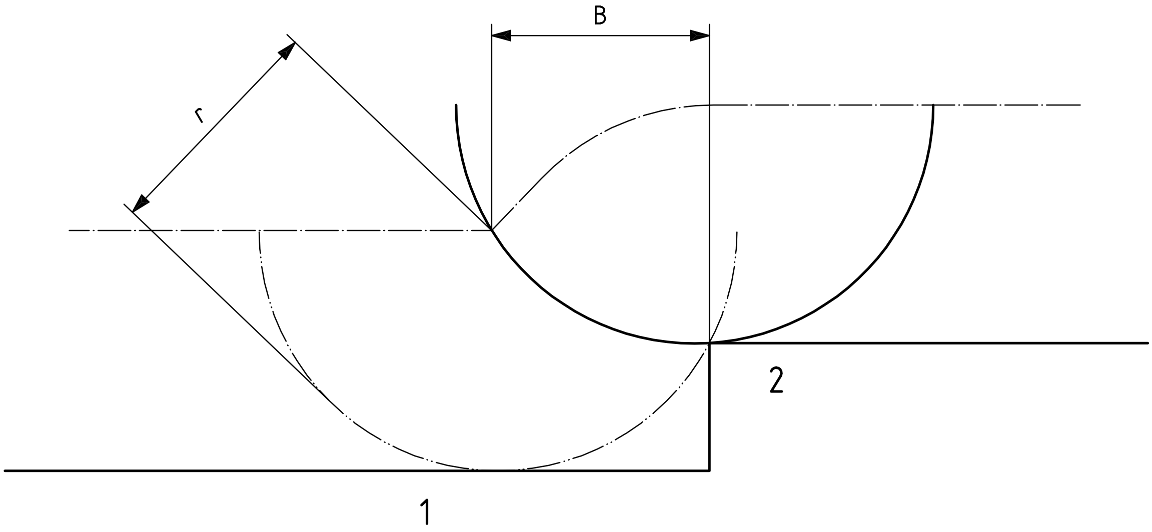
The contact-probe profilometer shall be in accordance with ISO 3274. The instrument shall be calibrated by using step-height calibration standards in accordance with the limits given in Clause 10.

In metrology, it is always very important to have calibration and checking conditions corresponding to the measurement conditions. Therefore, the calibration or certified reference materials should be as similar as possible to the step height to be measured.

6.2 Contact probe

The stylus chip of a contact probe is made of diamond, and has a conical shape. The vertical angle of the chip is 60° or 90°. The radius of the chip is any of 2 µm, 5 µm, 10 µm, and 12,5 µm.

NOTE The effect of the stylus-tip radius on the lateral profile resolution, i. e. the step-edge broadening due to the bluntness of the tip (see Figure 2), should be considered. However, due to the horizontal compression of the displayed picture (far greater vertical than horizontal magnification), this effect can be neglected for routine coating thickness measurements where definitive horizontal resolution is unimportant.



Key

1 substrate

2 step

Figure 2 — Broading of the step "B" due to the bluntness (radius r) of the stylus tip

7 Test pieces

7.1 General consideration

The test pieces shall comprise a fine ceramic thin film coated onto the surface of a substrate such as glass, or a piece cut from such an item.

The test pieces shall have dimensions sufficient to ensure stability on the test piece support of the profilometer.

Select a representative test specimen from the coating to be tested. Clean the specimen, using an appropriate method for the coating, so that it is free from dust, oil, moisture and any other surface films. The test-pieces may be produced by machining from a larger board on which the fine ceramic thin film was formed to the appropriate size.

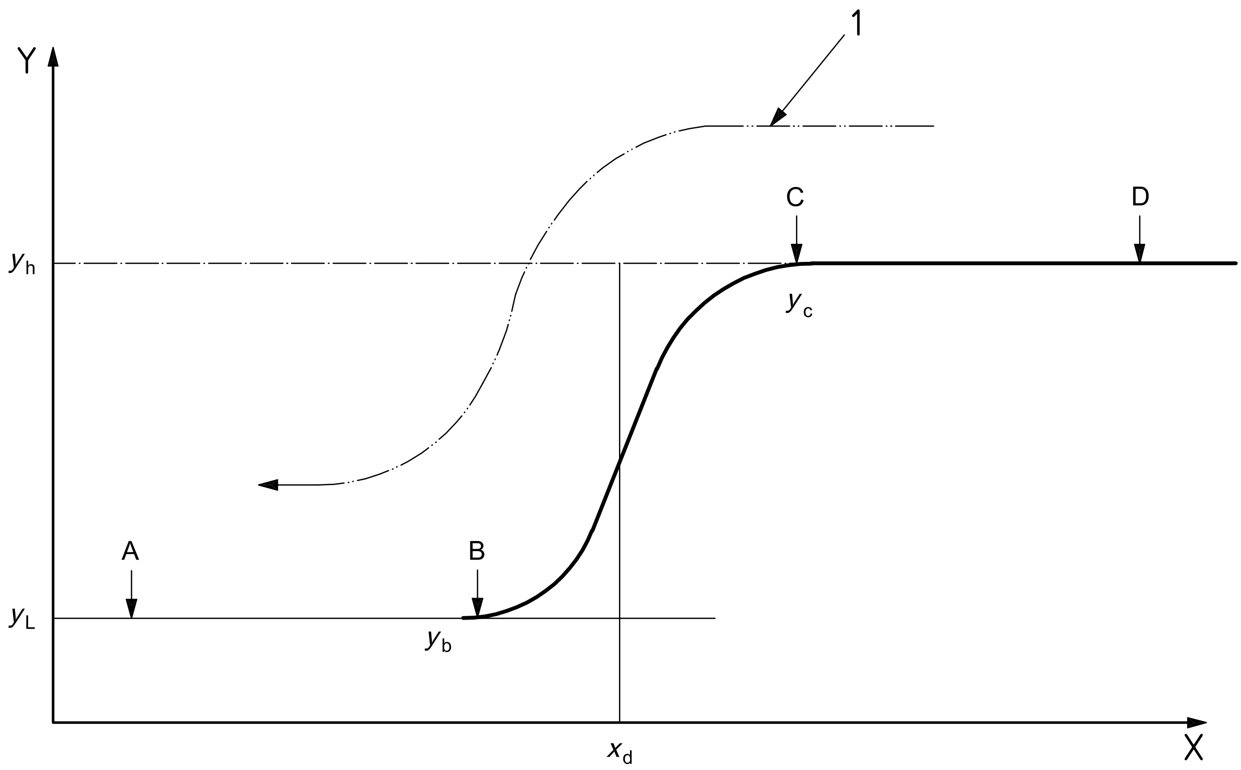
NOTE 1 The level difference is formed by taking away part of the film by chemical etching, or by depositing the film through a mask.

NOTE 2 When covering part of the substrate during deposition, it can happen that the deposition rate near the step is influenced by the covering medium. This results in a step which is not representative for the coating thickness. This can be prevented by using very thin covering plates, or it can be circumvented by etching away part of the coating after the coating process.

7.2 Surface conditions

Because surface roughness will affect the result, the Ra value of both substrate and coating shall be no greater than 1/5 of the step height, and the average wavelength of the roughness shall be $<$ 10 % of AB for the substrate or CD for the coating (see Figure 3).

NOTE The repeatability of the step-height measurement depends on the electronic noise level of the instrument, the digitization increment of the signal and the mechanical stability of the stylus or sample motion. However, the major source of statistical variation in calculated step heights is the surface roughness Ra of the step specimen, which is the ultimate uncertainty in assigning a height value to a stylus step profile.



Step height = $y_h - y_L$

Key

1 direction of travel of stylus

X Horizontal.

Y Vertical.

y_b y-position of point B

y_c y-position of point C

Figure 3 — Procedure for the determination of the coating step-height thickness

Systematic errors are related to non-parallel reference lines at both sides of the step either due to insufficiently flat substrates or to local coating thickness artefacts related to sample preparation.

7.3 Number of test pieces

At least three test pieces shall be used.

8 Procedure

8.1 Calibrate the contact-probe profilometer (6.1).

8.2 Level the sample so that the uncoated portion of the substrate is parallel to the x-axis datum of the measuring machine. Procedures for this will depend upon the machine being used. Errors in levelling will result in differences between the measured and the true value of the coating thickness (see Annex A). As shown in Annex A, the magnitude of the error depends on both the angular levelling error and the ratio width of the step/coating thickness.

8.3 Set the stylus loading to its lowest value (from 0,05 mN to 0,3 mN) and scan the step striding over the level difference. In order for the limits of the step width (points B and C in Figure 3) to be accurately identified, the travel for the probe on the uncoated and coated portions of the sample (distances AB and CD respectively; see Figure 3) shall each be a minimum of 10 $\times$ BC.

The contact probe should be scanned along a line perpendicular to the boundary between coated and uncoated portions of the substrate.

To avoid damage to the profile of the step, scanning shall take place in the direction from the coating surface to the substrate surface, i.e. down the step given in Clause 4.

NOTE It is desirable that the stylus loading is small so that it does not give rise to damage of the coating or the substrate. However, if the stylus loading is too small it may pick up external vibrations and produce an irregular profile.

8.4 Inspect the scan, for example by light microscopy, in order to determine whether any damage to the sample has occurred.

8.5 Repeat the procedure to give five measurements for each sample tested. Because the surfaces on either side of the step are often not flat and not parallel, a single coating step-height thickness measurement is not significant.

9 Calculation

Locate the x-position for the inflection point in the step. Calculate the two least-squares straight lines from representative points on either side of the step (see Figure 3). Calculate the difference in y-values of the two straight lines at the x-position previously determined for the inflection point. The obtained value is the step-height thickness of the coating.

If the data are not digitized (chart record), the drawing of the two least-squares straight lines may be done visually, upon agreement between the parties.

When substrate surface roughness and the average wavelength of the roughness are much smaller than the values specified in 7.2, the film thickness may be directly calculated from the difference of y_{b} and y_{c} (in Figure 3), upon agreement between the parties.

It is recommended that the plotted-out step height be at least 10 mm to enable accurate measurement of the step.

Only the representative data for the upper and lower level of the step should be considered to eliminate artifacts caused by the curvature of the step.

In the case of the standard measurement, 15 values of the film thicknesses are obtained from the three test pieces. The average of these 15 values given to three significant figures should be taken as the value. When the standard deviation is required, it should be calculated in the normal way using each of the 15 separate values and rounded to two significant figures.

10 Limits to step height

Using the method specified above, contact-probe profilometers can repeatedly identify steps in the 2 nm to 5 nm range and repeatedly measure larger steps. Height calibrations of contact-probe profilometers can be accurate to about 1 %, assuming that the standard is calibrated to an accuracy of better than 1 %.

NOTE In the case of measurement for the ceramics film with 100 nm thickness or less, it is difficult to obtain an accuracy of 0,1 nm. It is necessary to carry out special consideration and preparation.

11 Test report

The test report shall include the following information:

a) name of the testing establishment;

b) date of the test, report identification and number, signatory;

c) reference to this International Standard, i.e. “determined in accordance with ISO 18452”;

d) the name of certified reference materials used for calibration and checking the performance of the profilometer;

e) description of the test material; type of product, type of coating, date of receipt;

f) the test conditions employed, including stylus loading, stylus radius, magnification and length of measurement;

g) method of test specimen sampling and preparation;

h) method of calculation of step height (see Clause 9);

i) at least five values for the coating step-height thickness;

j) test results [film thickness, standard deviation (optional)];

k) comments about the test or the test results.

Annex A
(informative)

Effect of amplification factor and levelling error on measured layer thickness

In order to make small vertical displacements clearly visible, profilometers normally amplify displacements in the y direction more than in the x direction. This amplification can have a very significant effect upon the value of coating thickness obtained, depending upon how it is calculated. If it is assumed that the "amplification factor" for displacements in the y direction compared to those in the x direction is A, then it can be seen from Figure A.1 that the fractional error in the value of t (the coating thickness) resulting from the assumption that $(y_2 - y_1) = At$ (i.e. step height = amplification factor × true thickness) is:

$$1/At\,\{At[1 + (w/t)\,\tan\alpha]\cos\alpha - At\} = [1 + (w/t)\tan\alpha]\cos\alpha - 1$$

If $w < t\,\tan\alpha$, i.e. $w/ < t\,\tan\alpha$ then the step width, w, makes no contribution to the error and the fractional error in t resulting from the assumption that $(y_2 - y_1) = At$, is

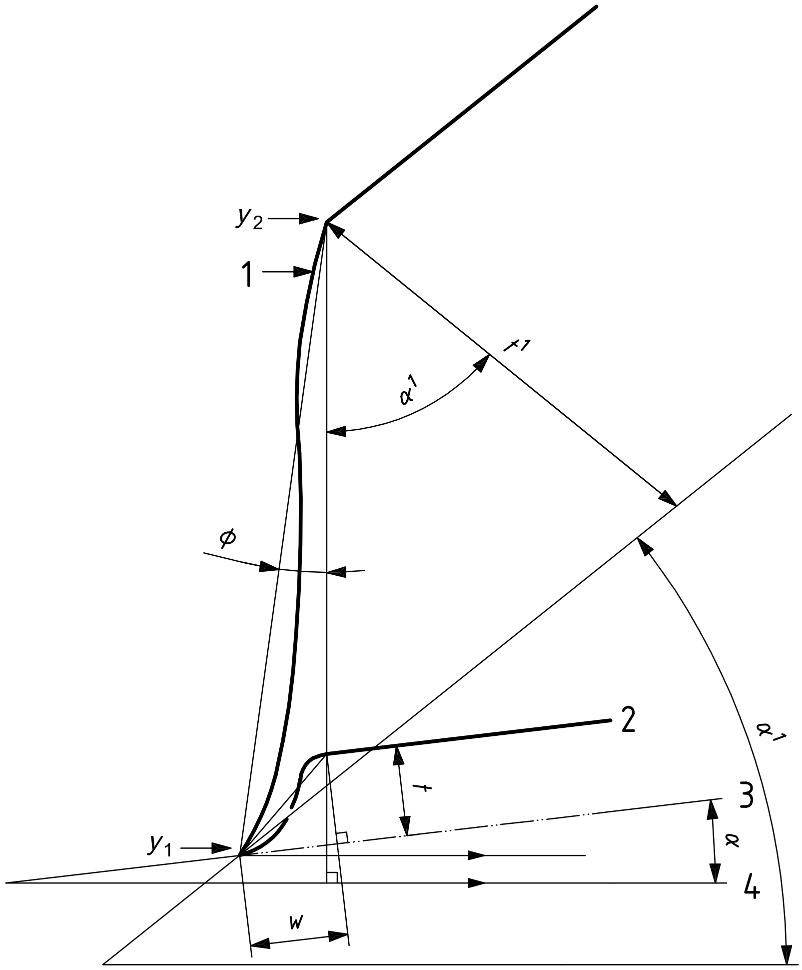
$$1/At[(At/\cos\alpha) - At] = [1/\cos\alpha) - 1 \quad \text{(A.1)}$$

For $w/t < t\,\tan\alpha$, i.e. for the case where the step width does contribute to the error in t, the fractional error in t is

$$[1 + (w/t)\tan\alpha]\cos\alpha - 1 \quad \text{(A.2)}$$

Equations (A.1) and (A.2) are plotted for various values of α and w/t on the accompanying graph (Figure A.2) from which the influence of the ratio (step width w):(step height t) can be clearly seen.

NOTE Because of the influence of A on the value of α^1 (see Figure A.1), large errors in the value of t can result from the use of t^1 (the perpendicular distance between the projection of the substrate and coating surfaces) to determine t, depending on the value of A.



$$(y_2 - y_1) = A(t + w\tan\alpha)\cos\alpha = At(1 + \frac{w}{t}\tan\alpha)\cos\alpha$$

$$t = \frac{(y_2 - y_1)}{\cos\phi}\cos(\alpha^1 + \phi)$$

Key

1 amplified step profile

2 surface of coating

3 surface of substrate

4 x-axis datum

α true inclination (levelling error) of substrate surface to x-axis datum

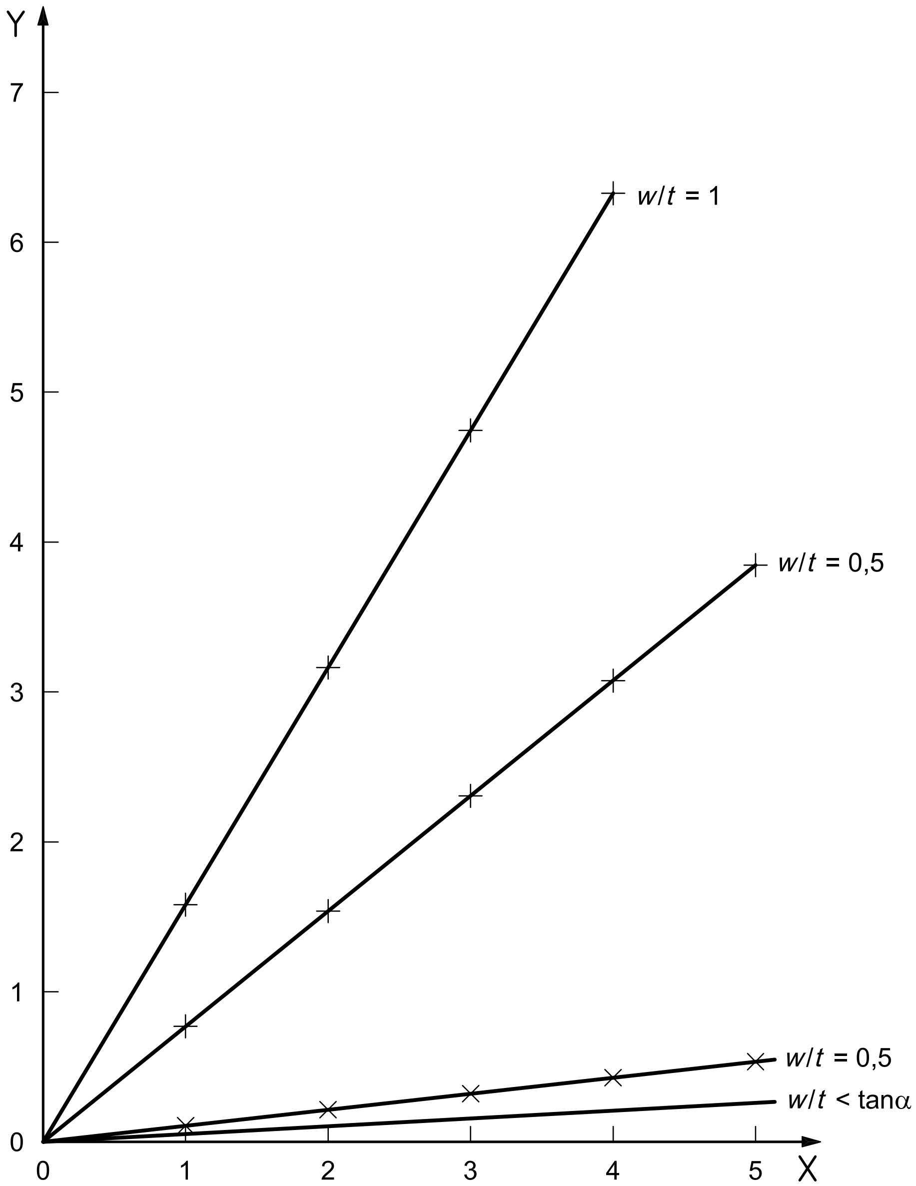
α^1 amplified inclination of “substrate surface” to x-axis datum

t^1 projection of coating thickness under amplification

w true step width

NOTE The other symbols are defined in the text of Annex A.

Figure A.1 — Effect of y-axis amplification on step profile



Key

Y percentage error in value of t, calculated from $t = (y_2 - y_1)/A$

X inclination, α (in degrees)

Figure A.2 — Graph showing percentage error in value of t, calculated from $t = (y_2 - y_1)/A$, as a function of inclination (levelling error) and the ratio (step width w): (step height t)

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