SLOVENSKI STANDARD
SIST ENV 1071-1:2000

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Advanced technical ceramics - Methods of test for ceramic coatings - Part 1: Determination of coating thickness by contact probe profilometer

Advanced technical ceramics - Methods of test for ceramic coatings - Part 1: Determination of coating thickness by contact probe profilometer

Hochleistungskeramik - Verfahren zur Prüfung keramischer Schichten - Teil 1: Bestimmung der Schichtdicke mit einem Kontaktprofilometer

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Céramiques techniques avancées - Méthodes d'essai pour revetements céramiques - Partie 1: Détermination de l'épaisseur du revetement par profilometre a contact

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ICS:

25.220.99	Druge obdelave in prevleke	Other treatments and coatings
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EUROPEAN PRESTANDARD

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Descriptors: Composite materials, reinforcing materials, ceramics, coatings, tests, determination, thickness, profilometer

English version

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CEN members are required to announce the existance of this ENV in the same way as for an EN and to make the ENV available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the ENV) until the final decision about the possible conversion of the ENV into an EN is reached.

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart,36 B-1050 Brussels

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SIST ENV 1071-1:2000

Contents list

iTeh STANDARD PREVIEW
(standards.iteh.ai)
SIST ENV 1071-1:2000
https://standards.iteh.ai/catalog/standards/sist/46312a97-34f1-45df-9d79-9ab5bf67e924/sist-env-1071-1-2000

SIST ENV 1071-1:2000

Foreword

This European pre-standard has been prepared by CEN/TC184 'Advanced technical ceramics'.

ENV 1071 has six Parts:

Part 1 : Determination of coating thickness by contact probe profilometer
Part 2 : Determination of coating thickness by cap grinding method
Part 3 : Determination of adhesion by a scratch test
Part 4 : Determination of chemical composition
Part 5 : Determination of the porosity
Part 6 : Determination of the topography

CEN/TC184 approved this European pre-standard by resolution 3/1992 during its fifth meeting held in Brussels on 1992-03-31.

In accordance with CEN/CENELEC Internal Regulations, the following countries are bound to announce this European pre-standard:

Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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SIST ENV 1071-1:2000
https://standards.iteh.ai/catalog/standards/sist/46312a97-34f1-45df-9d79-9ab5bf67e924/sist-env-1071-1-2000

SIST ENV 1071-1:2000

Advanced technical ceramics - Methods of test for ceramic coatings - Part 1: Determination of coating thickness by contact probe profilometer

1 Scope

This Part of ENV 1071 describes a method for the determination of the thickness of ceramic coatings by measurement of the step height using a contact probe profilometer.

NOTE : An alternative measurement of thickness, using a cap grinding method, is described in ENV 1071-2 .

2 Normative references

This European Pre-Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at appropriate places in the text and in the publications listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Pre-Standard only when incorporated in it by amendment of revision. For undated references the latest edition of the publication referred to applies:

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ENV 1071-2	Advanced technical ceramics - Methods of test for ceramic coatings - Part 2 : Determination of coating thickness by the cap grinding method

SIST ENV 1071-1:2000
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ENV 1071-3	Advanced technical ceramics - Methods of test for ceramic coatings - Part 3 : Determination of adhesion by a scratch test
ISO 1880:1979	Instruments for the measurement of surface roughness by the profile method - Contact (stylus) instruments of progressive profile transformation - Profile recording instruments
ISO 3274:1975	Instruments for the measurement of surface roughness by the profile method - Contact (stylus) instruments of consecutive profile transformation - Contact profile meters, system M
ISO 4287-1:1984	Surface roughness - Terminology - Part 1 : Surface and its parameters

3 Principle

The coating thickness often plays a major role in the performance of coated tools and machine parts. Many different techniques have been developed for assessing the coating thickness. Among these, the step height measurement is a very promising technique because of its easiness, wide applicability and accuracy.

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In order to create a step, part of the substrate has to remain uncoated. This can be done by covering part of the substrate during deposition, e.g. by means of a covering plate or tape or by scratching the coating substrate/system until delamination of the coating occurs (see ENV 1071-3).

The thickness of the coating can then be determined by scanning this step beneath the contact stylus of a contact probe profilometer (see figure 1).

NOTE : When covering part of the substrate during deposition, it may happen that the deposition rate near the step is influenced by the covering medium (e.g. local deformation of the plasma ball due to a covering plate). 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 instead of not covering part of the substrate.

In the case of line-or-sight processes (such as ion beam deposition methods), shadowing effects are to be avoided, in order to create a representative step.

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4 Apparatus

The contact probe profilometer shall be in accordance with ISO 1880:1979 or ISO 3274:1975 At present, commercially available apparatus has the following optimum capabilities:

SIST ENV 1071-1:2000
https://standards.iteh.ai/catalog/standards/sist/46312a97-34f1-45df-9d79-9ab5bf67e924/sist-env-1071-1-2000

a) vertical resolution : min 1Å (depending on the height range selected).
b) sampling interval : min 0,02 µm to max 5 µm (for lateral resolution, see clause 7).
c) height range : min 10 µm to max 300 µm.
d) scan length range : min 10 µm to max 400 mm.
e) scan speed : min 1 µm/s to max 25 mm/s.
f) data points per scan : min 2000 to max 65400.
g) stylus force : min 8 µN to max 1000 µN.
h) sample thickness : max 63,5 mm.

The stylus tip radius may be either 2,5 or 10 µm. The instrument shall be calibrated by using step height calibration standards in accordance with the limits given in clause 7.

NOTE 1 : In metrology, it is always very important to have calibration and checking conditions corresponding to the measurement conditions. Therefore, the calibration or check standards should be as similar as possible to the step height to be measured.

NOTE 2 : The effect of the diamond stylus tip radius on the lateral profile resolution, i.e. the step edge broadens due to the bluntness of the diamond 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.

SIST ENV 1071-1:2000

5 Sampling and preparation of test specimens

Select a representative test specimen from the coating to be tested. Clean the specimen so that it is free from dust and other particles, and also from oil or other surface films.

6 Procedure

Set the stylus loading to its lowest value in order. Scan the step and make tests in order to determine whether any damage to the sample has occurred.

NOTE 1 : In order to be able to use loadings less than 20 µN, the instrument should be mounted on a vibration isolation table.

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.

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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.

SIST ENV 1071-1:2000
https://standards.iteh.ai/catalog/standards/sist/4b3f4b12-...-9ab5bf67e924/sist-env-1071-1-2000

NOTE 2 : 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, or by the phenomenon of 'stylus flight' (see figure 4).

Because both surfaces of the step are often not flat and not parallel, a single coating step height thickness measurement is not significant. Therefore, repeat the procedure to give five measurements for each sample tested.

7 Limits

7.1 Step height

Using the method described above, contact probe profilometers can repeatedly identify steps in the 20-50A 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 %.

SIST ENV 1071-1:2000

7.2 Lateral resolution

The lateral resolution of surface features depends on both the stylus radius and the slopes of the surface features being profiled. For a specimen surface roughness R(a)(see ISO 4287-1:1984)of 0,1 µm, the expected lateral resolutions are:

a) for a stylus tip radius of 2 µm : 3 µm
b) for a stylus tip radius of 5 µm : 4,5 µm
c) for a stylus tip radius of 10 µm : 6 µm

NOTE : The best situation is to sample the profile with a sampling interval at least four times closer than the expected lateral resolution of the stylus.

8 Repeatability

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 R(a) of the step specimen, which is the ultimate uncertainty in assigning a height value to a stylus step profile.

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SIST ENV 1071-1:2000
https://standards.iteh.ai/catalog/standards/sist/46312a97-34f1-45df-9d79-9ab5bf67e924/sist-env-1071-1-2000

9 Test report

The test report shall include the following information:

a) The name of the testing establishment
b) Date of the test, report identification and number; signatory
c) A reference to this standard, i.e. determined in accordance ENV 1071-1
d) A description of the test material; type of product, type of coating, date of receipt
e) Method of test specimen sampling and preparation
f) Method of calculation of step height (see clause 6)
g) At least five values for the coating step height thickness
h) Comments about the test or the test results