



# SLOVENSKI STANDARD

## SIST ENV 1071-5:2000

01-december-2000

---

### Advanced technical ceramics - Methods of test for ceramic coatings - Part 5: Determination of porosity

Advanced technical ceramics - Methods of test for ceramic coatings - Part 5:  
Determination of porosity

Hochleistungskeramik - Verfahren zur Prüfung keramischer Schichten - Teil 5:  
Bestimmung der Porosität

Céramiques techniques avancées - Méthode d'essai pour revêtements céramiques -  
Partie 5: Détermination de la porosité

<https://standards.iteh.ai/catalog/standards/sist/fd3c9d66-907f-4cec-a11a-5a43fdbf2de/sist-env-1071-5-2000>

Ta slovenski standard je istoveten z: **ENV 1071-5:1995**

---

#### **ICS:**

25.220.99	Druge obdelave in prevleke	Other treatments and coatings
81.060.30	Sodobna keramika	Advanced ceramics

**SIST ENV 1071-5:2000**

**en**

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

SIST ENV 1071-5:2000

<https://standards.iteh.ai/catalog/standards/sist/fd3c9d66-907f-4cec-a11a-5a43fddb2de/sist-env-1071-5-2000>

EUROPEAN PRESTANDARD

ENV 1071-5

PRÉNORME EUROPÉENNE

EUROPÄISCHE VORNORM

February 1995

ICS 81.060.20

Descriptors: composite materials, ceramics, technical ceramics, protective coatings, tests, determination, porosity, metallography

English version

**Advanced technical ceramics - Methods of test for  
ceramic coatings - Part 5: Determination of  
porosity**

Céramiques techniques avancées - Méthode  
d'essai pour revêtements céramiques - Partie 5:  
Détermination de la porosité

Hochleistungskeramik - Verfahren zur Prüfung  
keramischer Schichten - Teil 5: Bestimmung der  
Porosität

**(standards.iteh.ai)**

SIST ENV 1071-5:2000

<https://standards.iteh.ai/catalog/standards/sist/fd3c9d66-907f-4ccc-a11a-5a43fddb2de/sist-env-1071-5-2000>

This European Prestandard (ENV) was approved by CEN on 1993-06-10 as a prospective standard for provisional application. The period of validity of this ENV is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the ENV can be converted into an European Standard (EN).

CEN members are required to announce the existence 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.

CEN members are the national standards bodies of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

**CEN**

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

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

© 1995

All rights of reproduction and communication in any form and by any means reserved in all countries to CEN and its members.

Ref. No. ENV 1071-5:1995 E

**Contents**

	<b>Page</b>
Foreword	3
1 Scope	4
2 Normative references	4
3 Definitions	4
4 Principle	5
5 Test instrument	5
6 Sample preparation	5
7 Procedure	6
8 Expression of results	8
9 Test report	8
Annex	9
A (Informative) : Bibliography	9

**ITeH STANDARD PREVIEW**  
**(standards.iteh.ai)**

[SIST ENV 1071-5:2000](https://standards.iteh.ai/catalog/standards/sist/fd3c9d66-907f-4cec-a11a-5a43fddb2de/sist-env-1071-5-2000)

[https://standards.iteh.ai/catalog/standards/sist/fd3c9d66-907f-4cec-a11a-](https://standards.iteh.ai/catalog/standards/sist/fd3c9d66-907f-4cec-a11a-5a43fddb2de/sist-env-1071-5-2000)

[5a43fddb2de/sist-env-1071-5-2000](https://standards.iteh.ai/catalog/standards/sist/fd3c9d66-907f-4cec-a11a-5a43fddb2de/sist-env-1071-5-2000)

## Foreword

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

ENV 1071 has five 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 porosity

CEN/TC 184 approved this European pre-standard according to resolution 1 taken during its eighth meeting held in London, United Kingdom, on 1993-06-10.

In accordance with the 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, United Kingdom.

(standards.iteh.ai)

SIST ENV 1071-5:2000

<https://standards.iteh.ai/catalog/standards/sist/fd3c9d66-907f-4cec-a11a-5a43fddb2de/sist-env-1071-5-2000>

## 1 Scope

This Part of ENV 1071 describes a method of test for characterization of micropores of ceramic coatings by means of metallographical examination. The method is applicable to thin ceramic coatings, such as produced by means of chemical and/or physical vapour deposition methods and related techniques.

NOTE : Coatings of this type are usually applied to protect the substrate from the environment and provide a surface having other beneficial properties, such as wear resistance, corrosion protection, special electrical and/or magnetic properties and decorative appearance.

Porosity is detrimental to the intended functioning of these coatings in that it provides openings through which the substrate may be corroded, leading to spalling of the coating.

## 2 Normative references

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

<https://standards.iteh.ai/catalog/standards/sist/fd3c9d66-907f-4ccc-a11a-5e43fd1b2de/sist-env-1071-5-2000>

EN 45001 : General criteria for the operation of testing laboratories

## 3 Definitions

For the purposes of this Part of ENV 1071, the following definitions apply:

**3.1 Pores:** volumetric spacings of various sizes in the material which are free from solid or liquid material.

**3.2 Closed pores:** pores which appear as isolated or clustered voids in the material and have no connection to the surface.

**3.3 Open pores:** pores which appear as isolated or clustered voids in the material and have no connection to the surface.

**3.4 Micropores:** closed or open pores which show dimensions only detectable at microscopic scales.

## 4 Principle

Porosity in the coating is determined by preparing an area of coating in plan and/or cross section of high microscope surface quality such that the area can be viewed using a light microscope or scanning electron microscope. Quantitative assessment of porosity is carried out using image analysis techniques which is either directly applied to the microscope or to photo-micrographs of the inspected coatings.

## 5 Test instrument

The test instrument shall be either:

- a) a light microscope, when pore sizes are greater than 2  $\mu\text{m}$ ;
- b) a scanning electron microscope (SEM), for all pore sizes.

NOTE 1 : Image analysis equipment capable of quantitative determination of both the number of pores and their area of fraction is preferred.

NOTE 2 : It is possible to count the number of pores directly and measure their area fraction using simple grids, but these methods are tedious and, therefore prone to error. Annex A (A.1 to A.6) lists some useful references which will assist in understanding image analysis methodology.

[SIST ENV 1071-5:2000](https://standards.iteh.ai/catalog/standards/sist/fd3c9d66-907f-4cec-a11a-5a43fddb2de/sist-env-1071-5-2000)

<https://standards.iteh.ai/catalog/standards/sist/fd3c9d66-907f-4cec-a11a-5a43fddb2de/sist-env-1071-5-2000>

## 6 Sample preparation

### 6.1 General

Light microscopical inspection needs specularly reflecting surface quality. For the assessment of closed porosity by means of light microscopy a polished cross section shall be prepared such that the plan of inspection shall range from the surface of the coating to the substrate interface.

For SEM analysis it is essential that the pores are distinguishable by means of electronic image contrast features. When inspecting hard brittle coatings, SEM analysis of micropores shall be verified on the unprepared surface and/or on fractured cross sectioned areas of the coating.

### 6.2 Sampling

Cut the sample from the component or test piece using procedures that minimize damage.

Record the positions within components or test pieces from which sections are prepared.

NOTE : Diamond or boron nitride saws have been found to be very effective in this purpose.

### 6.3 Mounting

The mounting medium shall be chosen to allow good edge retention and be of comparable hardness to the coating, in order to minimize difficulties during grinding and polishing.

NOTE : Normally a cold setting epoxy resin mounting material is recommended and, in order to obtain adequate hardness, a hard filler such as alumina powder can be used.

### 6.4 Grinding and polishing

For preparation of polished cross sections, it is necessary first to mount the selected region so that a flat polished area with minimal edge rounding is obtained.

NOTE : Edge retention can be improved by coating the outer surface of the sample with an additional layer such that if surrounding occurs, the area of interest remains unaffected. Electrodeless nickel plating or sputtering with a metal layer are commonly used.

Use standard metallographic procedures in which the initial stages of preparation are carried out with grinding by using progressively finer abrasive silicon carbide papers.

During grinding, examine the prepared area at each stage to ensure that all the damage from the previous stage has been removed. It is frequently possible with brittle coatings that large areas of coating may be damaged during the curing or grinding stages. In this case it is often necessary to prolong the grinding at any given stage (sometimes for as much as 30 min) in order to remove all such damage.

Polish with diamond paste down to 1  $\mu\text{m}$  grade. Further polishing with colloidal silica may be required to obtain a scratch free surface. After polishing, clean the sample in suitable solvents in an ultrasonic bath to remove all polishing debris.

## 7 Procedure

### 7.1 General

When examining a specularly reflecting sample, use the light microscope in the first instance to determine the size range of the porosity in the coating.

For examination in a SEM ensure that the sample is in good electrical contact with the specimen mounting block. For an electrically non-conducting sample, deposit a thin electronically conducting layer (carbon, gold, aluminium) on the surface, to avoid charge of the surface which would result in poor quality images.

NOTE : Some SEMs (Field Emission SEM) can be operated at low voltage (1 - 2 kV) where charging of the sample can be avoided, so that coating the sample with a conducting layer would not be necessary. The spatial resolution is diminished to a tenth in comparison with conventional SEM.



## 7.2 Measurement of porosity

7.2.1 Having selected the instrument (7.1), examine the coating to determine total porosity.

7.2.2 Focus the microscope on the area to be examined and optimize contrast conditions to clearly differentiate the pores in the image analysis equipment or photomicrographs. Choose the magnification for examination so that it is appropriate for the pore size and pore distribution under investigation.

NOTE : For pore size  $> 4 \mu\text{m}$  in diameter a magnification of a maximum 100 times is recommended, while for pores  $< 4 \mu\text{m}$  in diameter a magnification of a maximum 1000 times should be used.

7.2.3 Select representative areas from the coating such that at least 100 pores can be measured, but in any case measure a minimum of 10 frames or 10 photomicrographs. Choose these areas using random selection procedures to ensure unbiased results.

7.2.4 Measure each frame or photomicrograph to obtain a value of the number of pores and their area fraction. Make replicate (minimum three) measurements of each frame or photomicrograph to determine the repeatability of values.

7.2.5 When using manual methods, determine the number of pores per unit area by simply counting the number of pores in a given frame or photomicrograph and dividing by the area of the sample examined. When automatic methods are used, follow the equipment manufacturer's instructions when implementing the software.

7.2.6 For manual methods of determining pore area fraction, use an appropriately sized, calibrated grid and measure the number of grid intersections at each pore. Calculate the area fraction using the following relationship:

$$A = \frac{(x + x^1)}{y}$$

where

- A is the area fraction
- x is the number of intersections of the grid that fall within a pore
- $x^1$  is half the number of intersections of the grid that fall on a pore boundary
- y is the total number of grid intersections in the field of view

When automatic methods are used to determine pore area fraction, follow the equipment manufacturer's instructions when implementing the software.