TECHNICAL REPORT

ISO/TR 26946

First edition
2011-11-15

Standard method for porosity measurement of thermally sprayed coatings

Méthode normalisée de mesure de la porosité des revêtements obtenus par projection thermique

Reference number
ISO/TR 26946:2011(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TR 26946 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*.

Standard method for porosity measurement of thermally sprayed coatings

1 Scope

This Technical Report describes a method for characterizing the porosity of thermally sprayed coatings by metallographical examination.

This method is particularly applicable to oxide coatings, such as Al_2O_3, ZrO_2 and TiO_2, produced by plasma spray. It also considers the purposes to test the size, shape and density of pores for thermally sprayed coatings.

2 Purpose

The main purpose of porosity measurement is to determine the quality of a thermally sprayed coating and its freedom from porosity, particularly on those areas of the significant surface that demand a functional requirement.

This Technical Report provides a standard process that is suitable for determining the porosity of thermally sprayed coatings, as part of the total quality assurance programme.

This Technical Report is also intended to provide a standard way to present the porosity of thermally sprayed coatings.

3 Classification

The microstructure of a thermally sprayed ceramic coating is characterized by the existence of various pores, microcracks, splat boundaries and unmelted particles, because of the nature of the process. Although different terms are used, both the pores and the microcracks are volumetric spaces, which are free from coating material. The pores can be divided into closed pores, open pores and micropores. Closed pores appear as isolated clustered voids in the coating and have no connection with the surface; open pores appear as the same voids but have a connection with the atmosphere, either directly or from one pore to another; micropores are either closed or open pores which show dimensions only detectable on a microscopic scale. The difference between pores and microcracks lies mostly in their aspect ratios (ratio of the major axis over the minor axis), so, they are collectively treated as pores. The fraction of volumetric space covered by the pores in thermally sprayed coatings is defined as porosity.

4 Principle

The porosity of thermally sprayed coatings is determined by preparing an area of the inspected coating with a cross-section of high microscopic surface quality, which can be viewed using a light microscope or a scanning electron microscope (suggested). A quantitative assessment of the porosity of the inspected coatings is carried out by using an image analysis technique on the microscope.

5 Apparatus

The following equipment is necessary for the porosity measurement of thermally sprayed coatings.

5.1 Cut-off wheels (recommended) or **diamond wire** or **high pressure water-jet cutting equipment**, (according to equipment in existence) for sectioning coating samples to a proper size with minimal damage.

5.2 Cleaning apparatus, with ultrasonic equipment.

5.3 Mounting equipment.

5.4 Grinding and polishing equipment, (semi-automated or automated grinding/polishing machines are recommended for consistent reproducibility).

5.5 Scanning electron microscope (recommended) or **light microscope**, for viewing the inspected sample on a cross-section and obtaining digital images.

5.6 Computer, with analysing software for porosity evaluation on digital images.

All equipment should undergo regular maintenance and calibration to assure reliability and repeatability of the measurement. At the same time, all metallographic personnel should have the proper training to allow them to perform the required functions and analyses.

6 Metallographic preparation

6.1 General

Metallographic preparation of thermally sprayed coatings is critical for the porosity results. The requirements for detail and monitoring will vary from system to system, depending upon the degree of automation in the preparation. The basic steps for the preparation are given in 6.2 to 6.5.

6.2 Sectioning

If sectioning is required, two commonly used methods are abrasive wheel cutting or diamond wire cutting. The first one, which is comprised of a diamond or boron nitride saw (more effective in this purpose) that breaks down readily exposed fresh cutting surfaces, is usually best for a wide range of coatings. Sectioning should be done with the cutting force from coating to substrate and minimal clamping pressure on the sample. It will be better to secure the specimen for sectioning with a soft cushion, such as wood, if possible. The sectioning wheel should be as thin as possible to minimize damage, which must be removed in subsequent steps. Minimum pressure should be applied on the wheel to minimize possible overheating, with cooling by water if possible. The length of the test specimen should be greater than 1 cm. At least five test specimens should be taken from each sample in different positions.

6.3 Cleaning

Cleaning is an important step for removing all contaminants from the surface of the specimen. Three methods or any combination are recommended.

a) Washing samples with soap and water.

b) Brushing or soaking samples in solvent, such as acetone/alcohol, followed by application of heat treatment to drive off any internal absorption.

c) Cleaning samples by performing an initial/extra vacuum step (if using vacuum impregnation in mounting) to volatilize any entrapped materials.

6.4 Mounting

For the preparation of polished cross-sections, it is necessary to mount the selected region first so that a flat polished area with minimal edge rounding is obtained. In this case, edge retention can be improved by coating the outer surface of the sample with an additional layer during grinding and polishing. Electroless nickel plating or sputtering with a metal layer are commonly used. The mounting procedure/material depends on the following:

a) time available for mounting;

b) size of porosity and level of voids in the coating, and degree of interconnected porosity;

c) required viscosity of epoxy for impregnation of porosity is important (the viscosity of the cold-mount epoxy should be medium, especially when porosity in the coating is small and difficult to impregnate);

d) hardness of coating vs. mounting material. (The mounting medium should be chosen to allow good edge retention and be of comparable hardness to the coating, in order to minimize difficulties during grinding and polishing.)

Cold mounting, which can be assisted by heat, with vacuum impregnation alone and/or pressure impregnation is recommended.

6.5 Grinding and polishing

Generally, grinding and polishing parameters that must be considered/controlled in preparation are listed in Table 1. Additional care must be taken to remove cut-off damage during initial grinding if the sectioning step was used, and avoid over-polishing with colloidal silica in the final steps of preparation. During grinding, examine the prepared area at each stage to ensure that all the damage from the previous stage has been removed. In the case of polishing, the sample is polished with diamond paste down to 1 µm grade, then alumina paste is used with 0,3 µm 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. It should be noted that porosity evaluation is relatively a complicated process and grinding and polishing parameters should be chosen properly for reproducible porosity results. Typical procedures involving both grinding paper and disc formats are shown and suggested in Tables 2 and 3. These procedures will require modification for different coating types and equipment available in the specific laboratories. Semi-automatic/automatic machines in conjunction with written procedures that monitor/control critical parameters are recommended, which will result in consistent and reproducible results.

The kind and amount of consumables used in the metallographic process are obviously very critical to the final result. It is important to know the changes in consumable suppliers and these should be considered carefully. The specific trial samples should be run to assure similar performance and results, if changes have to be made to an already established procedure with new consumables.

Research should always be conducted to judge the preparation by SEM micrographs to confirm that no coarse feature occurs during metallographic preparation which is significant of the presence of pullouts, which inevitably result in deviation. Surface roughness is suggested as a crucial parameter to evaluate the quality of the preparation, which is connected with porosity range in the inspected coating and should be as low as possible.

Table 1 — Grinding and polishing parameters considered/controlled in preparation

Parameter	Description
Pressure	Load/mount area
Speed	Both table and specimen holder
Rotation direction	Relative rotation of head with respect to table
Format	Grinding disc vs. grinding papers Polishing: no-nap vs. high-nap clothes
Abrasive	Diamond, SiC, colloidal silica, Al_2O_3
Orientation	How samples are placed in holder with respect to wheel rotation
Frequency	How often is lubricant/abrasive applied
Kind of lubricant	Oil, water, alcohol
Quantity of lubricant	ml/min
Time	Processing duration for each step

Table 2 — Typical procedure with the grinding paper format

Surface	Grit size	Pressure	Speed	Time	Abrasive	Lubricant	Rotation
Grinding papers	180	40 kPa	300 rpm	10 min. (enough papers to flatten specimen and remove damage/edge effects)	SiC	Usually water	Complementary
Grinding papers	400,600 and 800	40 kPa	300 rpm	20 min.	SiC	Usually water	Complementary
Grinding papers	1000,1200 and 2000	40 kPa	300 rpm	30 min. (usually 2 papers per grit size)	SiC	Usually water	Complementary
No-nap cloth	Can be in the range of 1 to 6 µm diamond	40 kPa	300 rpm	Can be in the range of 2 to 4 min.	Poly- or mono-crystalline diamond	Usually water or alcohol	Complementary
Higher-nap cloth	Usually in the range of 0,3 to 0.5 µm	40 kPa	300 rpm	Usually 4 to 6 min.	Colloidal silica, Al_2O_3	Usually water or alcohol	Complementary

Table 3 — Typical procedure with the disc format

Surface	Grit size	Pressure	Speed	Time	Abrasive	Lubricant	Rotation
Fixed diamond or composite disc	40 to 60 µm	40 kPa	300 rpm	15 min.	Poly- or mono-crystalline diamond	Usually water	Complementary
Fixed diamond or composite disc	6 to 9 µm	40 kPa	300 rpm	30 min.	Poly- or mono-crystalline diamond	Usually water	Complementary
No-nap cloth	Can be in the range of 1 to 6 µm diamond	40 kPa	300 rpm	Can be in the range of 2 to 4 min.	Poly- or mono-crystalline diamond	Usually water or alcohol	Complementary
Higher-nap cloth	Usually in the range of 0.3 to 0,5 µm	40 kPa	300 rpm	Usually 4 to 6 min.	Colloidal silica, Al_2O_3	Usually water or alcohol	Complementary

7 Metallography procedure

It should be noted that metallographic examination is only meaningful for the well-prepared sample, as well as appropriate visual and numerical standards based on significant statistical analyses.

Pre-coating is necessary with a thin (10 to 20 nm) conducting film of carbon or gold (recommended) to avoid a change in the surface which would result in poor quality images.

Scanning electron microscopy (SEM) is strongly recommended as porosity result from optical microscopy is generally unreliable for the poor field depth, especially with higher magnification. Both SEI and BEI images can be employed depending on the metallographic preparation mentioned above. BEI image is recommended when no coarse feature such as pullout appears, which will be misunderstood as pores.

The magnification and the number of fields of view depend on both sample characters, such as porosity range and pore size distribution, which also depend on the deposition method and materials deposited, as well as measurement accuracy. It is contradictory to balance accuracy and the area of field of view. To achieve this goal, at least 15 fields of view at 1 000 × magnification, chosen randomly across the whole sample to ensure unbiased results, is recommended. Several more images may be necessary when porosity or its size distribution is high.

Focus the microscope on the area to be examined and optimize contrast conditions to distinguish the size and area of the pores clearly and suppress background variations in the image. The micrographs should be corrected prior to carrying out the analysis; features touching the image edges should be discarded for this purpose during the analysis.

It is very important to follow the manufacturer's instructions when implementing the software to determine the pore-area fraction. Generally, the pore edge in an image should be defined by a suitable threshold level. It is strongly recommended that the threshold level be adjusted by comparing the processed images with the original ones, in order to ensure that they are a reliable representation. It should be mentioned that this process should be carried out on each image unless they are obtained from the same SEM equipment and with the same parameters.

To increase the confidence in the measurements, statistical parameters, such as the mean diameter and standard deviation for a group of measurements, can be calculated.

8 Presentation of porosity

The porosity of a coating is an averaged value. To make sure that the averaged features of the entire coating can be represented by several small domains, the report of porosity from the statistical view is necessary.

Many statistical methods for evaluation of experimental data exist, such as probabilistic estimations, mean values, standard deviation (error). In practice a statistical treatment of experimental data is recommended for the presentation of porosity. In the case of a thermally sprayed coating, the real porosity should be predicted and presented from the finite measurement values as a mean value with standard deviation. The mean value comes from the porosity of each image, while the standard deviation depends on what kind of distribution the porosity of each image agrees with. As normal distribution always appears on porosity in thermally sprayed coatings, the porosity of a thermally sprayed coating should be presented as:

$$P\left\{\overline{P} \pm t_{\alpha/2}(n-1)\frac{S^*}{\sqrt{n}}\right\} \tag{1}$$

where

n is the number of images chosen for porosity;

P_i is the porosity of each image;

$\overline{P}$ is the average porosity of all images;

S^* is the standard deviation;

$t_{\alpha/2}(n\text{-}1)$ is the value of t distribution with flexibility n-1 and reliability 1-α;

$t_{\alpha/2}(n\text{-}1)$ can be obtained from a standard list (see Annex A).

The average porosity and the standard deviation can be obtained from:

$$\overline{P} = \frac{1}{n}\sum_{i=1}^{n} P_i \tag{2}$$

$$S^* = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}\left(P_i - \overline{P}\right)^2} \tag{3}$$

9 Test report

The test report should contain at least the following information:

a) the name of the testing establishment;

b) the date of the test;

c) a reference to this Technical Report, i.e. determined in accordance with ISO/TR 26946:2011;

d) description of the test material, type of products, type of coating, substrate, coating procedure, date of receipt;

e) test method;

f) specimen sampling, preparation, dimensions;

g) measurement equipment, measurement method, software used;

h) test results, total porosity, pore density and pore-area fraction;

i) number of replicated tests;

j) comments on the test and/or the test result.