TECHNICAL REPORT

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Standard method for porosity measurement of thermally sprayed coatings

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

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Foreword

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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.

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is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

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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.

Annex A
(informative)

List of t_α values with different n and α values

Table A.1 — List of t_α values with different n and α values

n	α = 0,25	α = 0,10	α = 0,05[a]	α = 0,025[a]	α = 0,01[a]	α = 0,005
1	1,000 0	3,077 7	6,313 8	12,706 2	31,820 7	63,657 4
2	0,816 5	1,885 6	2,920 0	4,302 7	6,964 6	9,924 8
3	0,764 9	1,637 7	2,353 4	3,182 4	4,549 7	5,840 9
4	0,740 7	1,533 2	2,131 8	2,776 4	3,746 9	4,604 1
5	0,726 7	1,475 9	2,015 0	2,570 6	3,364 7	4,032 2
6	0,717 6	1,439 8	1,943 2	2,446 9	3,142 9	3,707 4
7	0,711 1	1,414 9	1,894 6	2,364 6	2,998 0	3,499 5
8	0,766 4	1,396 8	1,859 5	2,306 0	2,896 5	3,355 4
9	0,702 7	1,383 0	1,833 1	2,262 2	2,821 4	3,249 8
10	0,690 8	1,372 2	1,812 5	2,228 1	2,763 8	3,169 3
11	0,697 4	1,363 4	1,795 9	2,201 0	2,718 1	3,105 8
12	0,695 5	1,356 2	1,782 3	2,178 8	2,681 0	3,054 5
13	0,693 8	1,350 2	1,770 9	2,160 4	2,650 3	3,012 3
14	0,692 4	1,345 0	1,761 3	2,144 8	2,624 5	2,976 8
15	0,691 2	1,340 6	1,753 1	2,131 5	2,602 5	2,946 7
16	0,690 1	1,336 8	1,745 9	2,119 9	2,583 5	2,920 8
17	0,689 2	1,333 4	1,739 6	2,109 8	2,566 9	2,898 2
18	0,688 4	1,330 4	1,734 1	2,100 9	2,552 4	2,878 4
19	0,687 6	1,327 7	1,729 1	2,093 0	2,539 5	2,860 9
20	0,687 0	1,325 3	1,724 7	2,086 0	2,528 0	2,845 3
21	0,686 4	1,323 2	1,720 7	2,079 6	2,517 7	2,831 4
22	0,685 8	1,321 2	1,717 1	2,073 9	2,508 3	2,818 8
23	0,685 3	1,319 5	1,713 9	2,068 7	2,499 9	2,807 3
24	0,684 8	1,317 8	1,710 9	2,063 9	2,492 2	2,796 9
25	0,684 4	1,316 3	1,708 1	2,059 5	2,485 1	2,787 4
26	0,684 0	1,315 0	1,705 6	2,055 5	2,478 6	2,778 7
27	0,683 7	1,313 7	1,703 3	2,051 8	2,472 7	2,770 7
28	0,683 4	1,312 5	1,701 1	2,048 4	2,467 1	2,763 3
29	0,683 0	1,311 4	1,699 1	2,045 2	2,462 0	2,756 4
30	0,672 8	1,310 4	1,697 3	2,042 3	2,457 3	2,750 0
31	0,682 5	1,309 5	1,695 5	2,039 5	2,452 8	2,744 0
32	0,682 2	1,308 6	1,693 9	2,036 9	2,448 7	2,738 5
33	0,682 0	1,307 7	1,692 4	2,034 5	2,444 8	2,733 3
34	0,681 8	1,307 0	1,690 9	2,032 2	2,441 1	2,728 4
35	0,681 6	1,306 2	1,689 6	2,030 1	2,437 7	2,723 8
36	0,681 4	1,305 5	1,688 3	2,028 1	2,434 5	2,719 5
37	0,681 2	1,304 9	1,687 1	2,026 2	2,431 4	2,715 4
38	0,681 0	1,304 2	1,686 0	2,024 4	2,428 6	2,711 6
39	0,680 8	1,303 6	1,684 9	2,022 7	2,425 8	2,707 9
40	0,680 7	1,303 1	1,683 9	2,021 1	2,423 3	2,704 5
41	0,680 5	1,302 5	1,682 9	2,019 5	2,420 8	2,701 2
42	0,680 4	1,302 0	1,682 0	2,018 1	2,418 5	2,698 1
43	0,680 2	1,301 6	1,681 1	2,016 7	2,416 3	2,695 1
44	0,680 1	1,301 1	1,680 2	2,015 4	2,414 1	2,692 3
45	0,680 0	1,300 6	1,679 4	2,014 1	2,412 4	2,689 6
∞	0,674	1,282	1,645	1,96	2,326	2,576

[a] More popularly employed.

Annex B
(informative)

Report of an international round robin test on the determination of porosity in plasma sprayed ceramic coatings by using image analysis of metallographically prepared cross sections

B.1 Introduction

This is a report of an international round robin test conducted within ISO/TC 107/WG 1 "Thermal spraying" on the determination of porosity in plasma sprayed ceramic coatings by using image analysis of metallographically prepared cross sections. It was agreed to conduct such test at the TC107 plenary meeting in Helsinki in 2007 under the leadership of the project leader, Prof. S.W. Lee.

B.2 Scheme of the round robin test

B.2.1 Participants

The following 8 laboratories from 6 countries participated in the round robin test.

China	Shanghai Ceramic Institute (SCI)
Korea	Sun Moon University (SMU)
Japan	National Institute for Materials Science (NIMS)
	National Institute for Advanced Industrial Science and Technology (AIST)
	Kurashiki Boring Kiko (KBK)
Finland	VTT
	TKK
Germany	N.N.
Poland	IMP

B.2.2 Sample preparation

Five kinds of ceramic coatings were prepared by 3 laboratories as shown in Table B.1. These samples were distributed among the participating laboratories. For the 8 % Y_2O_3-ZrO_2 coatings prepared at NIMS, an intentionally long spray distance of 200 mm was employed in order to produce samples with high porosity. Other coatings were sprayed by standard conditions at each laboratory to produce denser coatings.

Table B.1 — List of ceramic coatings prepared for round robin test

Organization	SunMoon University		VTT		NIMS
Material	Al_2O_3	TiO_2	Al_2O_3	TiO_2	8YSZ
Spray process	APS	APS	HVOF	HVOF	APS
Expected porosity	Low	Low	Low	Low	High

B.2.3 Guideline of procedures for porosity measurement

The following guideline about the metallographic preparation of coating cross sections, SEM observation and image processing was provided by the project leader, Prof. S.W. Lee.

To obtain reliable results on porosity measurement for thermal sprayed coatings, the following procedures are suggested.

B.2.3.1 Sectioning

Diamond saw is employed for cutting sample with the cut force from coating to substrate to a proper size (10 mm × 10 mm). For a minimal clamping pressure on sample, a soft wood cushion is used to secure the specimen for sectioning. The sectioning wheel is as thin as possible to minimize damage. A 2 kg load is applied on the wheel at speed of 12 m/min. with cooling by water. It is also possible to cut the impregnated sample for a higher strength to minimize damage on the inspected specimen.

B.2.3.2 Cleaning

The cut specimen with a proper size is soaked in acetone solvent for 10 minutes followed by alcohol solvent in an ultrasonic bath for 5 minutes to clear any entrapped materials during sectioning.

B.2.3.3 Mounting

The cleaned sample with a proper size is cold mounted by a cold-setting resin based on two fluid epoxy components with the dimension, typically ∅ 21 mm × 20 mm, at room temperature with vacuum impregnation for 30 minutes.

B.2.3.4 Grinding and polishing

The mounted sample is grinded by SiC paper with grit size 200, 400, 600, 800, 1200, and 2000 step by step lubricated with water at circling speed of 300 m/min. Two minutes is spent on stages with grit size of 200 and 400, 5 minutes on 600 and 800, while 15 minutes on 1200 and 2000. It should be kept in mind that damage in the former stage has to be removed in each stage. In the case of polishing, sample is polished with diamond paste from 3 to 1,5 to 1 µm grade with speed of 300 m/min for 5, 10, 15 minutes, then alumina paste with 0,3 µm grade for 20 minutes. After the polishing, clean the sample in alcohol solvents in an ultrasonic bath to remove all polishing debris.

B.2.3.5 Metallographic procedure

The treated specimen is pre-coated with a thin (10 to 20 nm) conducting film of gold to avoid charge of the surface before SEM observation. The specimen is observed by SEM on cross section with magnification of 100× to obtain the initial information such as thickness. 15 images at 1000× magnification, chosen randomly across the whole sample are obtained and saved. On these images, features touching the image edges are discarded for purpose during the analysis.

B.2.3.6 Porosity measurement by digital image analysis

The SEM images are used to calculate porosity measurement by digital image analysis with an image analysis software. The analysis includes two steps: Firstly, get a suitable threshold level, which is pre-adjusted and determined by a reliable representation comparing original and processed images. Secondly, calculate porosity by the image analysis software with the right threshold level obtained before. For each coating, the analysis should be repeated 15 times with different images obtained at 1000× magnification. Then, the mean porosity and corresponding deviation can be obtained.

B.3 Results and discussion

B.3.1 Initial results

The initially reported data were summarized in Table B.2. By looking at the average values from all the laboratories, it is clear that the 8YSZ sample contained the highest porosity around 10 vol% while the porosity values obtained with other samples were in the range from 2 to 4 vol%. When one looks at the maximum deviation in the reported values, i.e., Max-Min, the scatter is greater than or close to the average values, which means that the procedure adopted was not sufficiently reproducible. The data reported by one laboratory that used an optical microscope to image the cross sections have not been included for analysis since the data were generally out of the range (too large) from the data reported by the other laboratories that used SEM.

Table B.2 — Summary of porosity data reported by the participating laboratories

	APS			HVOF	
Sample	Al_2O_3 (SMU)	TiO_2 (SMU)	YSZ (NIMS)	Al_2O_3 (NIMS)	TiO_2 (VTT)
Average	2,42	2,29	9,63	3,85	2,50
STDV	0,82	1,37	2,48	2,67	1,14
Max-Min	2,60	4,70	7,59	6,86	2,45
Number of data reported	11	11	8	5	5

Figures B.1, B.2 and B.3 show the data from each laboratory for the three samples prepared by plasma spraying. It is not obvious if there exists specific trends based on some laboratories.

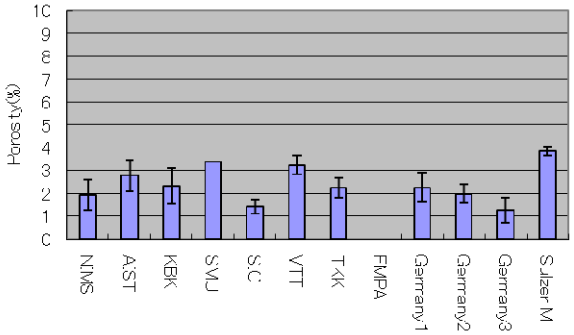

Figure B.1 — Reported porosity data for Al_2O_3 coatings prepared by SMU

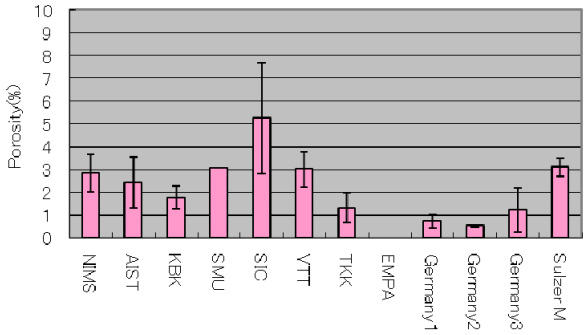

Figure B.2 — Reported porosity data for TiO_2 coatings prepared by SMU

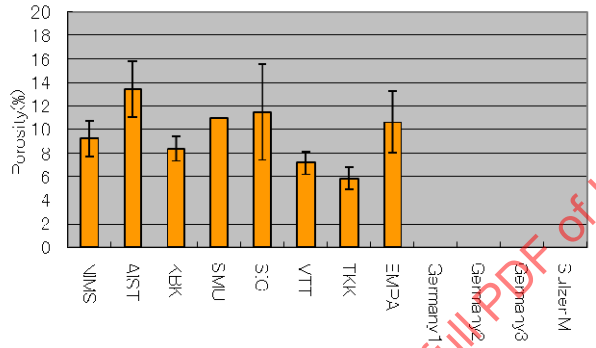

Figure B.3 — Reported porosity data for 8YSZ coatings prepared by NIMS

B.3.2 Second round test carried out in Japan

Since there were three laboratories participated in Japan, they carried out some additional experiments to find out what could be the reasons for the large scatters in the data they obtained from presumably the same samples.

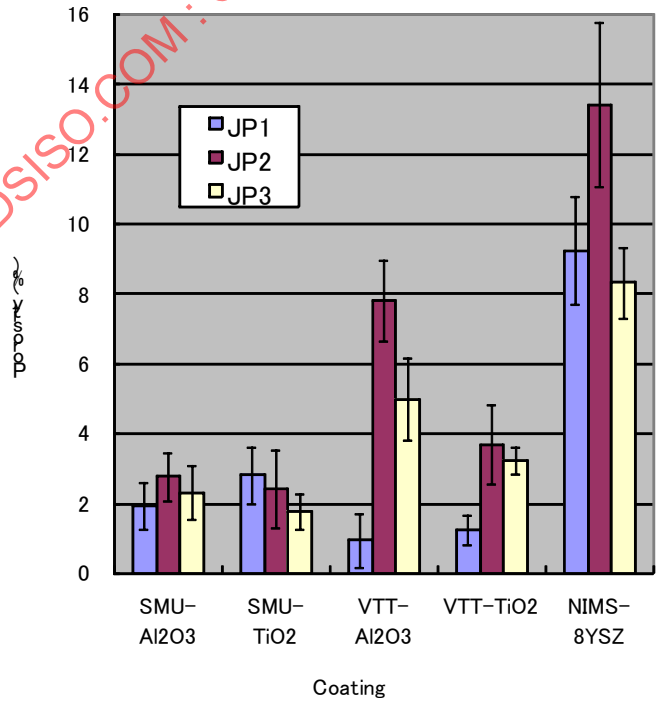

Figure B.4 — Data reported from the three Japanese laboratories in the first round

Figure B.4 shows the data reported from the three Japanese laboratories. One can notice that the porosity values for the two SMU samples were consistent whereas large variations were observed in the VTT and NIMS samples. In order to find out if the large differences observed in the first round were due to the image processing or other reasons such as surface preparation, the SEM images of the samples with large scatter, i.e., VTT-Al_2O_3 and NIMS-8YSZ, were exchanged among the three laboratories and analyzed by each laboratory's image processing software in the same way as done in the first round.

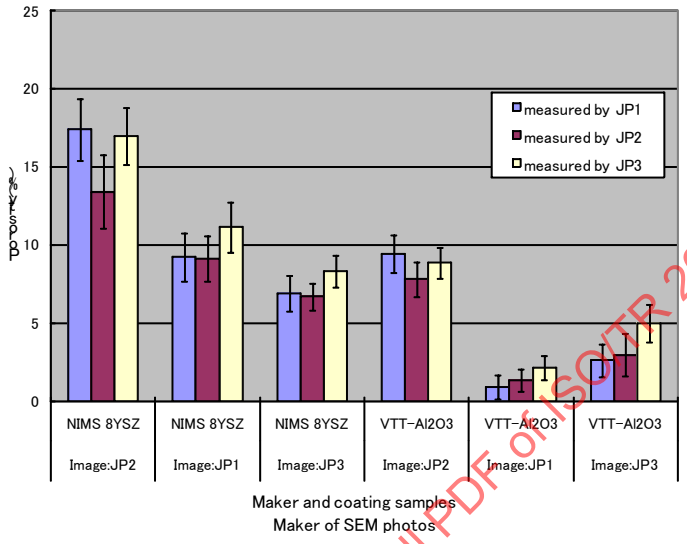

Figure B.5 — Results of SEM image exchange among the three Japanese laboratories

The results of SEM image exchange shown in Figure B.5 reveals that the difference due to the different image analysis software among the three laboratories was not so large and hence cannot be held responsible for the significant differences in the measured porosity data shown in Figure B.4. It should be noted, however, that there seems to exist some bias between the image processing among the three laboratories in the low porosity range below 5 % (JP3 > JP2 > JP1).

As the next step, it was decided to bring all the samples to JP1 laboratory and investigate their surfaces under its SEM.

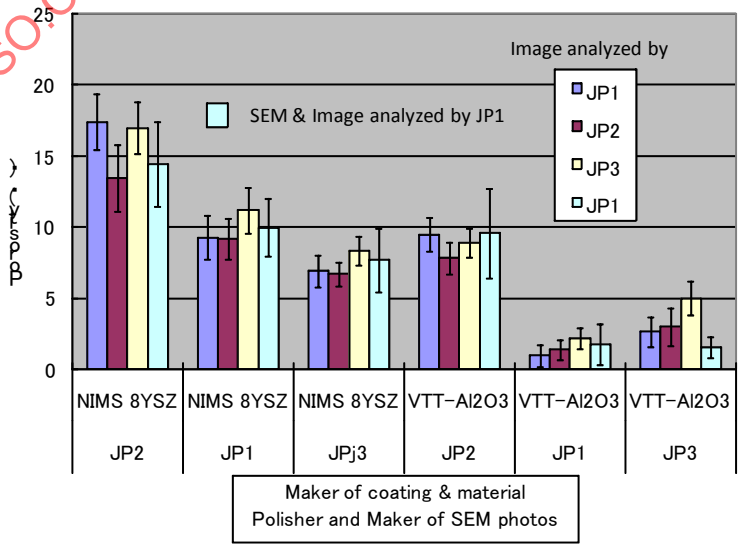

Figure B.6 — Results of SEM imaging and image analysis at JP1 and comparison with the results of image analysis at other laboratories

The results in Figure B.6 clearly shows that observation by the SEM at JP1 laboratory on the 3 different cross sections of each sample resulted in comparable values with those analyzed at other laboratories. These results of image exchange and recapturing by single SEM verify that SEM and image analysis were not the primary reason for the significant scatter of the porosity data shown in Figure B.4. The prepared cross sections from the same sample actually exhibited very different surfaces.

Then, the metallographic procedures in the three laboratories were examined. JP2 reported the highest porosity for VTT Al_2O_3, VTT TiO_2, and NIMS 8YSZ. It was realized that the contact pressure during grinding and polishing was greatly different among the three laboratories.

JP1: 45 kPa, JP2: 2～9 kPa, JP3: 14～17 kPa

In order to clarify the effects of the contact pressure, VTT Al_2O_3 and NIMS 8YSZ samples originally ground/polished by JP2 were further ground/polished by JP1 under the contact pressure at JP1, which was 5 lbs/sample, i.e., 45 kPa.

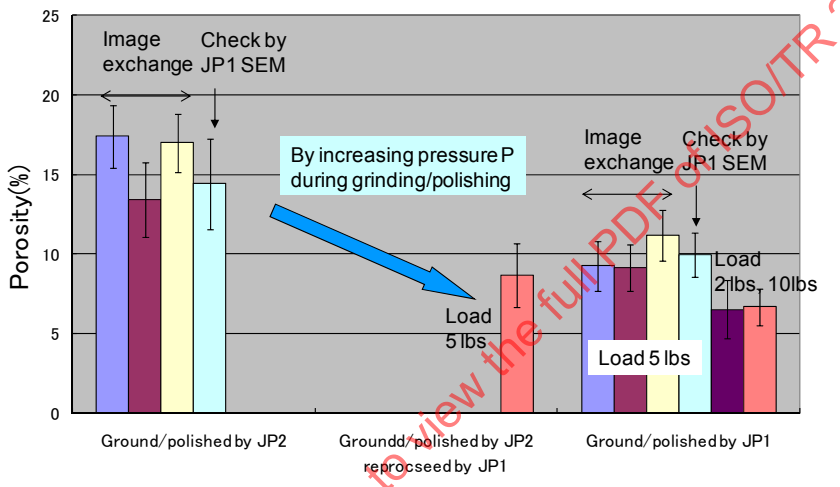

Figure B.7 — The cross section of a NIMS-8YSZ sample originally prepared at JP2 was reprocessed at JP1 at a higher contact pressure of 5 lbs/sample. The observed porosity decreased significantly to a comparable range reported from JP1 and JP3

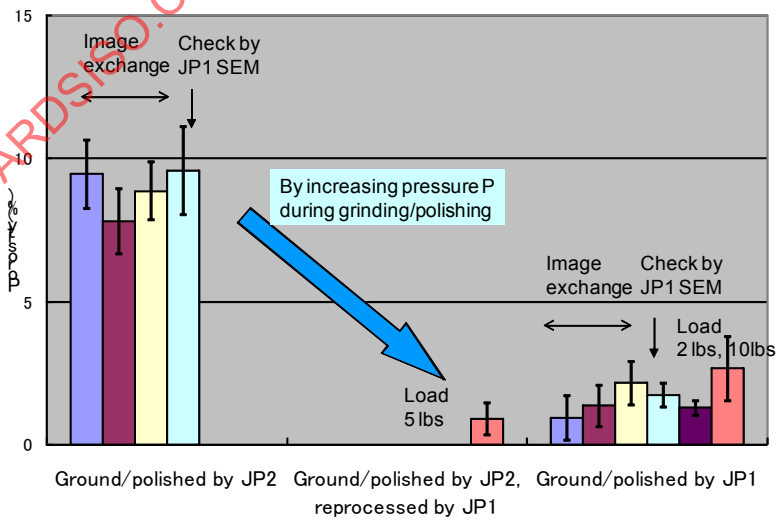

Figure B.8 — The cross section of a VTT-Al_2O_3 sample originally prepared at JP2 was reprocessed at JP1 at a higher contact pressure of 5 lbs/sample. The observed porosity decreased significantly to a comparable range reported from JP1 and JP3

Figures B.7 and B.8 show the results of reprocessing of the same samples originally prepared with lower contact pressures at JP2 by a higher contact pressure of 45 kPa at JP1. They both clearly indicate that the porosity on the cross section remarkably decreased. It is believed that the damage due to cutting had not been fully removed in JP2 due to the insufficient contact pressure at the initial grinding stage.

B.4 Reassessment of the round robin tests by the grinding/polishing conditions

The small study in Japan invoked an interest to check the results of the international round robin test in terms of the grinding/polishing conditions more carefully. It was recognized also that in the guideline provided by the project leader, there was a lack of specification about the contact pressure during grinding/polishing, which was shown to be a possible cause to generate significant differences on the prepared surface.