



Test Methods for Determining Area Percentage Porosity in Thermal Sprayed Coatings¹

This standard is issued under the fixed designation E 2109; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover procedures to perform porosity ratings on metallographic specimens of thermal sprayed coatings (TSCs) prepared in accordance with Guide E 1920 by direct comparison to standard images and via the use of automatic image analysis equipment.

1.2 These test methods deal only with recommended measuring methods and nothing in them should be construed as defining or establishing limits of acceptability for any measured value of porosity.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 3 Practice for Preparation of Metallographic Specimens²

E 7 Terminology Relating to Metallography²

E 562 Practice for Determining Volume Fraction by Systematic Point Count²

E 1245 Practice for Determining the Inclusion Content or Second-Phase Constituent Content of Metals by Automatic Image Analysis²

E 1920 Guide for Metallographic Preparation of Thermal Sprayed Coatings²

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods refer to Terminology E 7.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *halo effect*—unwanted detection of the perimeter of one phase (due to a shared gray value at the phase boundary) when setting the detection limits of another.

3.2.2 *linear detachment, n*—a region within a TSC in which two successively deposited splats of coating material have not

metallurgically bonded.

3.2.3 *porosity, n*—cavity type discontinuities (voids) or linear detachments within a sprayed coating.

3.2.4 *splat, n*—an individual globule of thermal sprayed material that has been deposited on a substrate.

4. Significance and Use

4.1 TSCs are susceptible to the formation of porosity due to a lack of fusion between sprayed particles or the expansion of gases generated during the spraying process. The determination of area percent porosity is important in order to monitor the effect of variable spray parameters and the suitability of a coating for its intended purpose. Depending on application, some or none of this porosity may be tolerable.

4.2 These test methods cover the determination of the area percentage porosity of TSCs. Method A is a manual, direct comparison method utilizing the seven standard images in Figs. 1-7 which depict typical distributions of porosity in TSCs. Method B is an automated technique requiring the use of a computerized image analyzer.

4.3 These methods quantify area percent porosity only on the basis of light reflectivity from a metallographically polished cross section. See Guide E 1920 for recommended metallographic preparation procedures.

4.4 The person using these test methods must be familiar with the visual features of TSCs and be able to determine differences between inherent porosity and oxides. The individual must be aware of the possible types of artifacts that may be created during sectioning and specimen preparation, for example, pullouts and smearing, so that results are reported only on properly prepared specimens. Examples of properly prepared specimens are shown in Figs. 8-10. If there are doubts as to the integrity of the specimen preparation it is suggested that other means be used to confirm microstructural features. This may include energy dispersive spectroscopy (EDS), wavelength dispersive spectroscopy (WDS) or cryogenic fracture of the coating followed by analysis of the fractured surfaces with a scanning electron microscope (SEM).

5. Apparatus

5.1 *Test Method A*—Test Method A requires a reflected light metallurgical microscope, upright or inverted, equipped with suitable objectives and capable of projecting an image onto a

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² *Annual Book of ASTM Standards*, Vol 03.01.

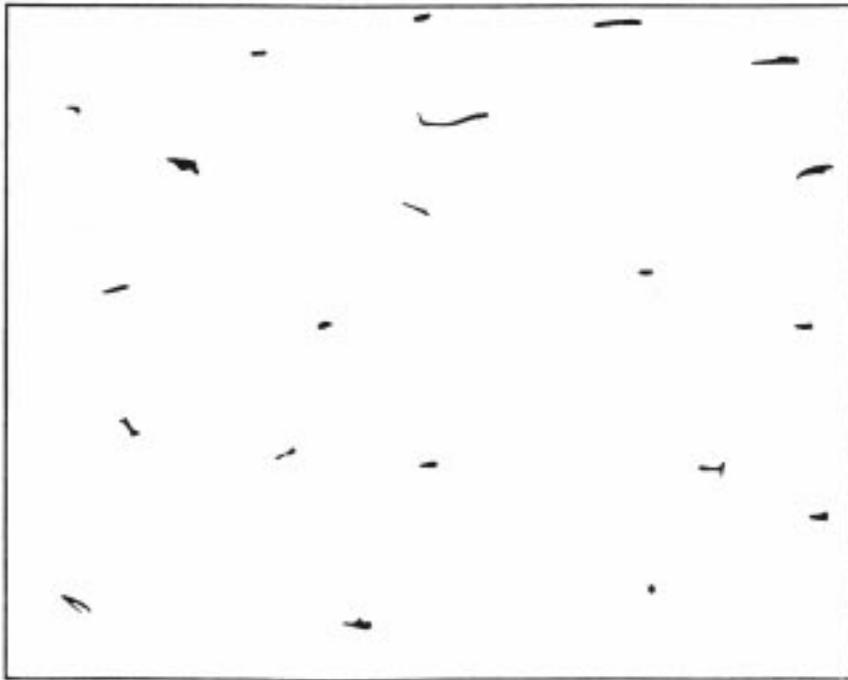


FIG. 1 — 0.5 % Porosity

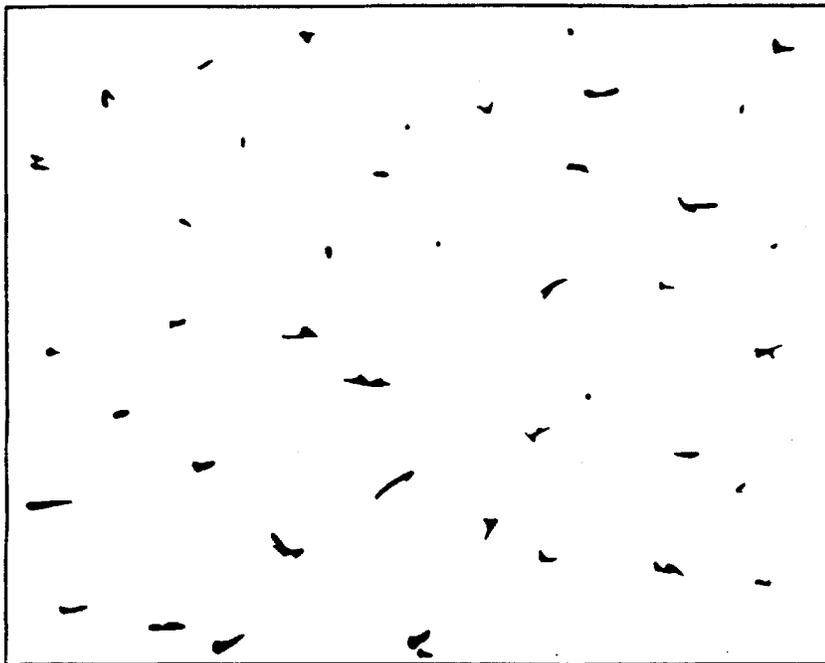


FIG. 2 — 1.0 % Porosity

ground glass viewing screen, video monitor or image recording media, such as film or video prints.

5.2 *Test Method B*—Test Method B requires a reflected light metallurgical microscope, upright or inverted, equipped with suitable objectives and interfaced to a video/digital image capture and analysis system. The microscope may be equipped with an automatic or manual stage. The use of an automated

stage should reduce operator fatigue.

5.3 *General Considerations*—The work area housing the test equipment must be kept relatively clean. This will minimize contamination of the specimen surface by dust that may settle on the polished surface of the specimen and influence the test results. In addition, adequate temperature and humidity controls must be in place to meet the computer or microscope

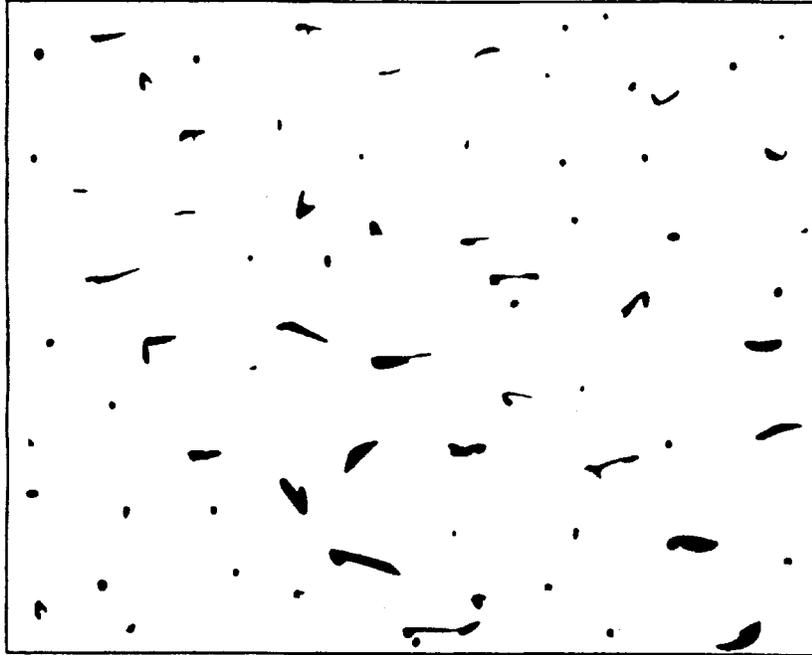


FIG. 3 — 2.0 % Porosity

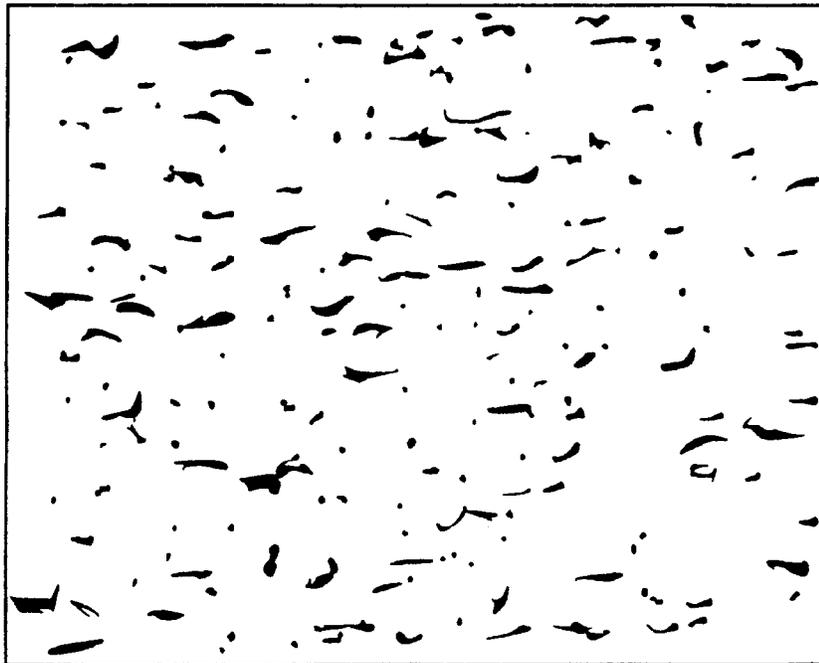


FIG. 4 — 5.0 % Porosity

manufacturer's specifications.

6. Sampling

6.1 Producer and purchaser shall agree upon the location and number of test specimens. Specimens may be metallographically sectioned from actual production pieces or from test panels comprised of representative substrates with identical production spraying parameters.

6.2 The specimens are metallographically prepared to reveal a polished plane through the test panel or part that is deemed critical. Specimens should include approximately 25 mm (1.0 in.) of coating length.

6.3 Multiple specimens may be selected to determine the homogeneity of the coating sprayed on the test panel or part.

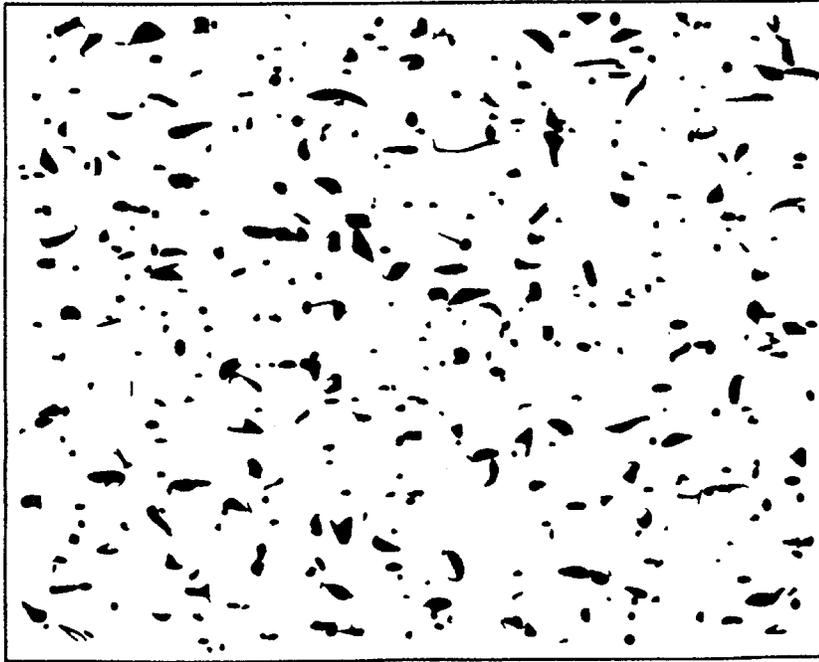


FIG. 5 — 8.0 % Porosity

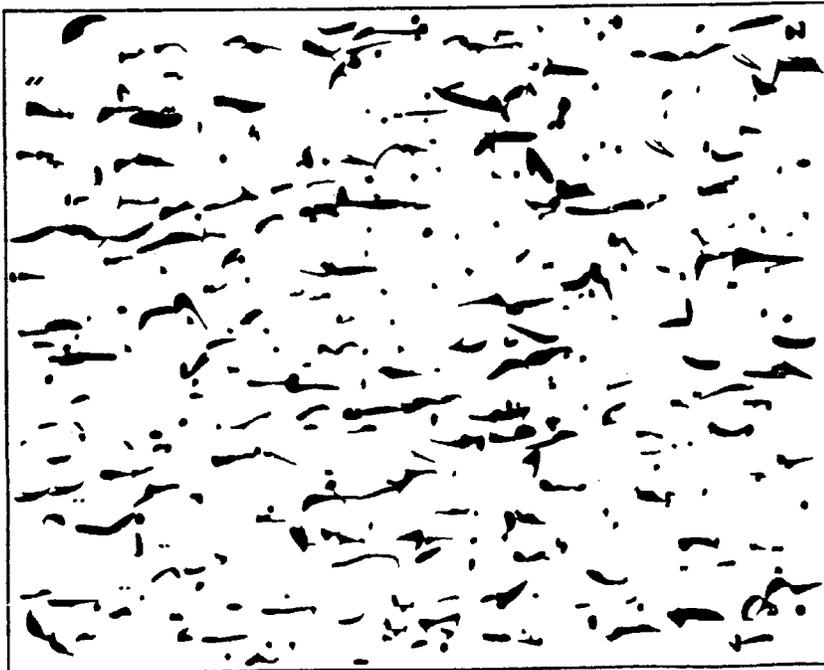


FIG. 6 — 10.0 % Porosity

For example, one may choose to sample from top-middle-bottom or edge-center-edge locations.

7. Specimen Preparation

7.1 Incorrect metallographic preparation of thermal sprayed specimens may cause damage to the coating or produce artifacts on the polished surface that may lead to biased

analytical results. The polished surface must reveal a clear distinction between inherent porosity, foreign matter, scratches and oxides. Polishing must not alter the true appearance of the inherent porosity by excessive relief, pitting pullout, or smearing.

7.2 General metallographic specimen preparation guidelines

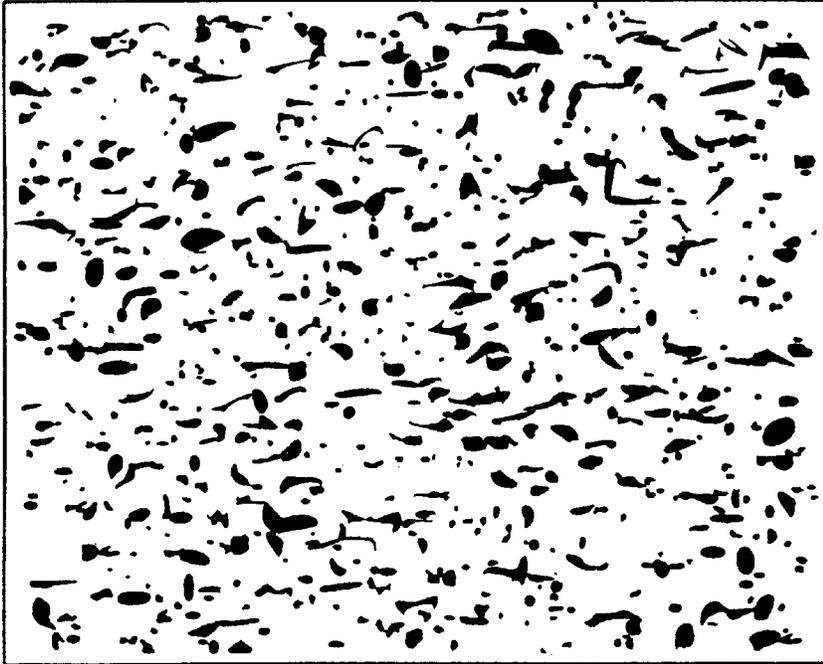
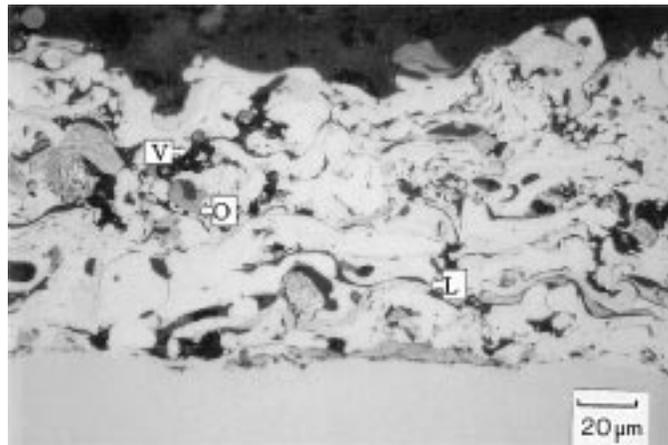


FIG. 7 — 15.0 % Porosity



NOTE 1—V = void, O = oxide, L = linear detachment

FIG. 8 Ni/Al TSC—500X

and recommendations are given in Practice E 3; however, manual metallographic preparation methods are not recommended for TSCs.

7.3 Use of automatic grinding and polishing equipment is recommended. Specific information regarding the preparation of TSCs using automated techniques is addressed in Guide E 1920.

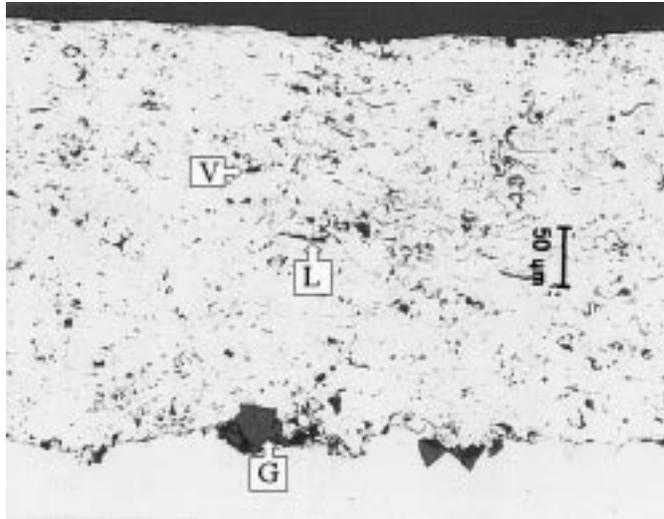
7.4 Damage to a brittle, porous TSC during specimen preparation is minimized when the specimen is vacuum impregnated with a low viscosity epoxy. The epoxy mounting compound fills the surface connected porosity and adds support to the coating during preparation.

7.5 Use of a dyed epoxy or fluorescent additive may be

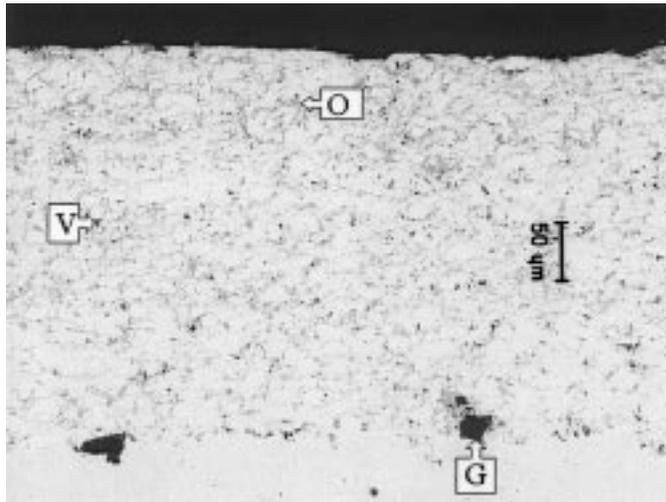
helpful in microstructural interpretation^{3,4}. Depending on the additive, a treated epoxy will fluoresce or appear as a distinct color when viewed with the appropriate light microscopy technique. This can eliminate any ambiguities concerning oxide content or pull-outs. Excitation and emission filters, darkfield illumination or polarized light may be required to reveal the color created by the dye or pigment. Consult the manufacturer's directions for the proper use of these materials.

³ Street, K.W. and Leonhardt, T.A., "Fluorescence Microscopy for the Characterization of Structural Integrity," *NASA Technical Memorandum 105253*, 1991.

⁴ Geary, A.R., "Metallographic Evaluation of Thermal Spray Coatings," *Microstructural Science*, Vol 19, D. A. Wheeler, et. al., eds., IMS and ASM Intl., Materials Park, OH, 1992, pp. 637-650.



NOTE 1—V = void, G = embedded grit, L = linear detachment
FIG. 9 Monel TSC—200X



NOTE 1—V = void, O = oxide, G = embedded grit
FIG. 10 Alloy 625 TSC—200X

8. Test Procedure

8.1 Test Method A (Direct Comparison):

8.1.1 This test method utilizes the images in Figs. 1-7 for comparison to microscopic fields of view on a polished specimen. Each figure has been assigned a value representing varying degrees of porosity.

8.1.2 Place the properly prepared specimen on the microscope stage and divert the image to a ground glass viewing screen or video monitor. Alternately, it may be recorded as a hard copy print.

8.1.3 Select a magnification that allows resolution of the voids and best fills the screen with the entire coating thickness. Often, a compromise must be reached whereby the entire coating thickness is not visible but a reduction in magnification would jeopardize the resolution of voids. It is more important to resolve all voids that contribute significantly to the total porosity area percentage. During this analysis the operator

must be able to distinguish the difference between oxides and epoxy infiltrated into voids.

8.1.4 Compare the image on the screen with Figs. 1-7. The image of interest and the figures should be approximately the same size. A minimum image area of 9 by 11 cm (3.5 by 4.5 in.) is required. This is the image size of a typical 4 by 5 in. instant print. One may either mask the viewing screen or alter the size of the figures (enlarge on a copier for instance) to achieve this requirement.

8.1.5 Record the value of the figure that most resembles the image of the present field of view. If the image does not closely match a figure, it may be rounded to the nearest whole number between figures values. For example, if the porosity in the current field of view falls between Figs. 4 and 5 representing porosity values of 5.0 % and 8.0 % respectively, a 6.0 or 7.0 may be recorded as appropriate.

8.1.6 If a field of view exhibits less than 0.5 % porosity, as

depicted in Fig. 1, it shall be reported as < 0.5 . These fields should be considered zero when computing the average area percentage porosity for the specimen.

8.1.7 If any single field has more porosity present than depicted in Fig. 7 that field shall be recorded as Outside Range (OR) along with a numerical value denoting the operator's estimate of the area percentage porosity. For example, a field thought to contain 25.0 % porosity should be recorded as: OR-25.

8.1.8 Using the same magnification, continue the procedure outlined above and record a value for at least ten random or contiguous fields. Do not overlap or re-measure fields of view.

8.1.9 If photomicrographs are used for comparisons, at least ten prints representing distinct fields of view at the same magnification are required. Do not overlap or re-photograph fields of view.

8.1.10 The point counting techniques in E 562 may be employed if direct comparison proves too difficult or to corroborate a Test Method A result.

8.2 Test Method B (Image Analysis):

8.2.1 Place the properly prepared specimen on the microscope stage and direct the image to the viewing screen. Guidelines for setting up a microscope and image analysis system including thresholding and interferences are given in Practice E 1245.

8.2.2 Select a magnification that allows resolution of the voids and best fills the screen with the entire coating thickness. If some of the substrate or mount is visible on the screen it must be masked in a manner that eliminates it from the total area used to calculate the area percentage porosity. Often, a compromise must be reached whereby the entire coating thickness is not visible but a reduction in magnification would jeopardize the detection of significantly sized voids. It is more important to resolve all voids that contribute significantly to the total porosity area percentage.

8.2.3 Once the best magnification has been determined, adjust the microscope's aperture and field diaphragms for the best resolution and contrast, saturate the light according to manufacturer's instructions for the image analysis system and, if necessary, incorporate the appropriate shading corrector for the objective in use.

8.2.4 Next threshold the porosity in the field of view. Thresholding, or image segmentation, is the process of selecting the appropriate range of gray values used to create a binary image. When thresholding the porosity, take care not to detect any oxides or other features close to the porosity's threshold limits.

8.2.5 Often, coating/oxide interfaces will begin to be detected when thresholding the porosity. This is referred to as the halo effect. To minimize this interference a binary editing function, such as masking, sieving or chord sizing may be used. Again, refer to the manufacturer's instructions for ways to eliminate small, unwanted features.

8.2.6 Alternately, a common binary image processing function known as opening may be used. Opening is a two step process (erosion and dilation) in which a layer of pixels is removed from the perimeter of each object represented in the binary image and then a layer of pixels is added back to the

perimeter of any remaining objects. The net effect is that very small and very thin objects can be entirely removed from the image while large objects will remain and retain near original dimensions.

8.2.7 Care must be taken not to significantly alter the area percentage porosity whenever employing any binary image processing functions.

8.2.8 The use of alternative microscopy techniques, for example, darkfield, polarized light or fluorescence, is permitted to facilitate thresholding of porosity that has been filled with a dyed or treated epoxy.

8.2.9 After a thresholding and image processing routine has been developed, check several fields of view to ensure that the porosity detection is correct.

8.2.10 Analyze at least 20 separate fields of view either in a random pattern or contiguously being careful not to overlap a previous field.

8.2.11 Do not incorporate any routine or technique that eliminates coating features that are touching the border of an image or guard frame.

8.2.12 If specimens are to be compared, one should use the same objective lens and instrument settings.

9. Statistical Analysis

9.1 No determination of porosity can be an exact measurement. Many specimens vary measurably in porosity from one field of view to another, this variation being responsible for a major portion of the uncertainty. Thus, no determination is complete without also calculating its precision within normal confidence. In accordance with common engineering practice, this section assumes normal confidence to represent the expectation that the actual error will be within the stated uncertainty 95 % of the time. Therefore, the following statistical determinations are required for results generated via Test Method B. Test Method A results are exempt from statistical determinations beyond the mean, maximum and minimum porosity values because they are based strictly on direct comparison.

9.2 After the desired number of fields have been measured, calculate the mean value of area percentage porosity according to:

$$\bar{X} = \frac{\sum X_i}{n} \quad (1)$$

where:

X_i = represents an individual value,

\bar{X} = the mean, and

n = the number of measurements.

9.3 Calculate the standard deviation of the individual measurements according to the usual equation:

$$S = \left(\frac{\sum (X_i - \bar{X})^2}{n - 1} \right)^{\frac{1}{2}} \quad (2)$$

where:

S = the standard deviation.

9.4 Calculate the 95 % confidence interval, 95 % CI, of each measurement according to:

$$95 \% CI = \frac{t \cdot s}{\sqrt{n}} \quad (3)$$

Table 1 lists values of t as a function of n .

9.5 Calculate the percent relative accuracy, % RA , of the measurements by dividing the 95 % CI value by the mean and expressing the results as a percentage, that is:

$$\% RA = \frac{95 \% CI}{\bar{X}} \cdot 100 \quad (4)$$

9.6 If the % RA is considered to be too high for the intended application, more fields should be measured and the calculations in 9.2-9.5 should be repeated. As a general rule, a 10 % RA (or lower) is considered to be acceptable precision for most purposes.

10. Report

10.1 Report the following information for each specimen:

10.1.1 Test method used;

10.1.2 Specimen identification;

10.1.3 Operator;

10.1.4 Date;

10.1.5 Magnifying power and numerical aperture of the objective used;

10.1.6 Total magnification used;

10.1.7 Calibration factor (when using Test Method B);

10.1.8 Mean, minimum and maximum area percentage porosity;

10.1.9 Standard deviation, 95 % confidence interval, and percent relative accuracy value; and,

10.1.10 Number of fields measured.

10.1.11 A histogram representing the above also may be included.

11. Precision and Bias

11.1 In general, the precision and bias of porosity measurements on TSCs depend on how well the specimens selected represent the actual coating and the metallographic preparation of those specimens. If the porosity varies greatly within a product, due to factors such as specimen geometry or fluctuations in the spraying process, specimen and field selection must adequately sample this variation.

11.2 Specimen preparation in accordance with Guide E 1920 will minimize test variability due to preparation techniques.

11.3 Improper setting of the threshold ranges for detection and discrimination of the porosity will bias results. If the detection or image processing scheme appears to be inadequate, the operator should abort the run and reset the threshold levels.

11.4 The presence of dust or other debris on the specimen surface or lenses of the imaging system will bias results towards higher values.

11.5 The choice of magnification can influence test results. The same objective lens should be used for all measurements of specimens within the same lot. Choose a magnification that allows discrimination of the pores that significantly contribute to the overall porosity value.

12. Keywords

12.1 area fraction; automatic image analysis; porosity; TSCs

TABLE 1 95 % Confidence Interval Multipliers, t (Eq 3)

No. of Fields, n	t	No. of Fields, n	t
20	2.093	27	2.056
21	2.086	28	2.052
22	2.080	29	2.048
23	2.074	30	2.045
24	2.069	40	2.020
25	2.064	60	2.000
26	2.060	∞	1.960

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