INTERNATIONAL

Designation: E2109 − 01 (Reapproved 2021)

Standard Test Methods for Determining Area Percentage Porosity in Thermal Sprayed Coatings[1]

This standard is issued under the fixed designation E2109; 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 E1920 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E3 Guide for Preparation of Metallographic Specimens

E7 Terminology Relating to Metallography

E562 Test Method for Determining Volume Fraction by Systematic Manual Point Count

E1245 Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis

E1920 Guide for Metallographic Preparation of Thermal Sprayed Coatings

[1] These test methods are under the jurisdiction of ASTM Committee E04 on Metallography and are the direct responsibility of Subcommittee E04.14 on Quantitative Metallography.

Current edition approved Sept. 1, 2021. Published November 2021. Originally approved in 2000. Last previous edition approved in 2014 as E2109 – 01(2014). DOI: 10.1520/E2109-01R21.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods refer to Terminology E7.

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 E1920 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

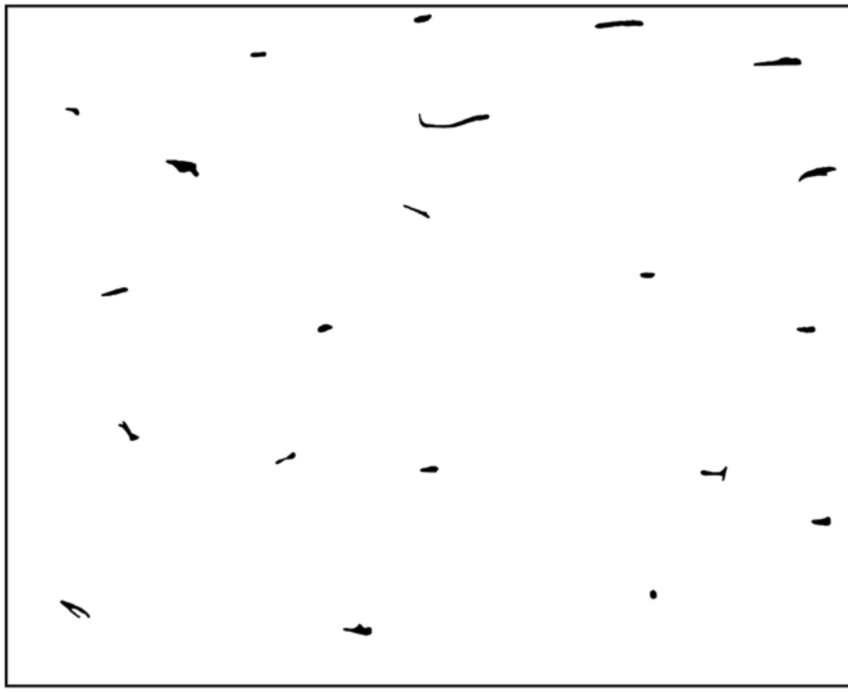


FIG. 1 — 0.5 % Porosity

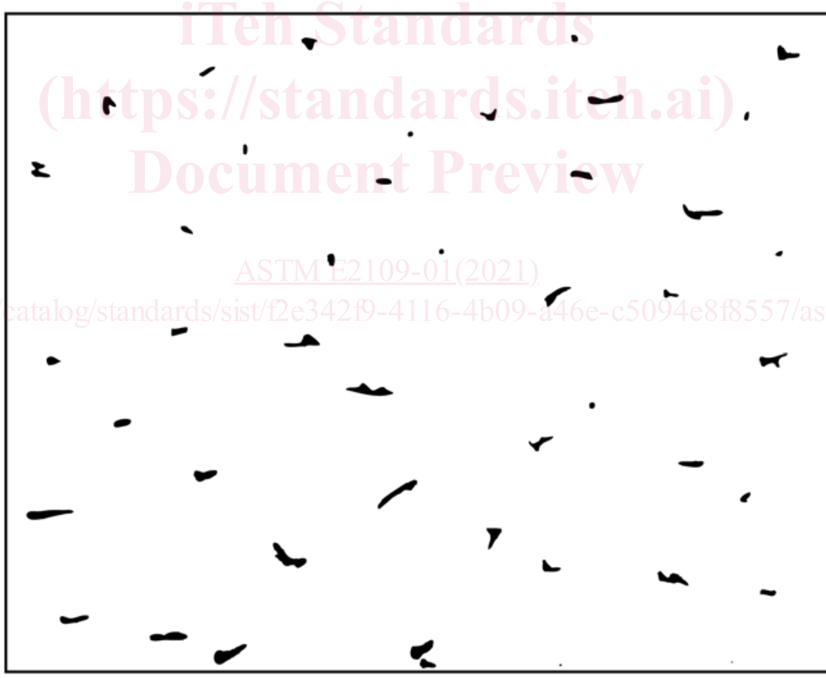


FIG. 2 — 1.0 % Porosity

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

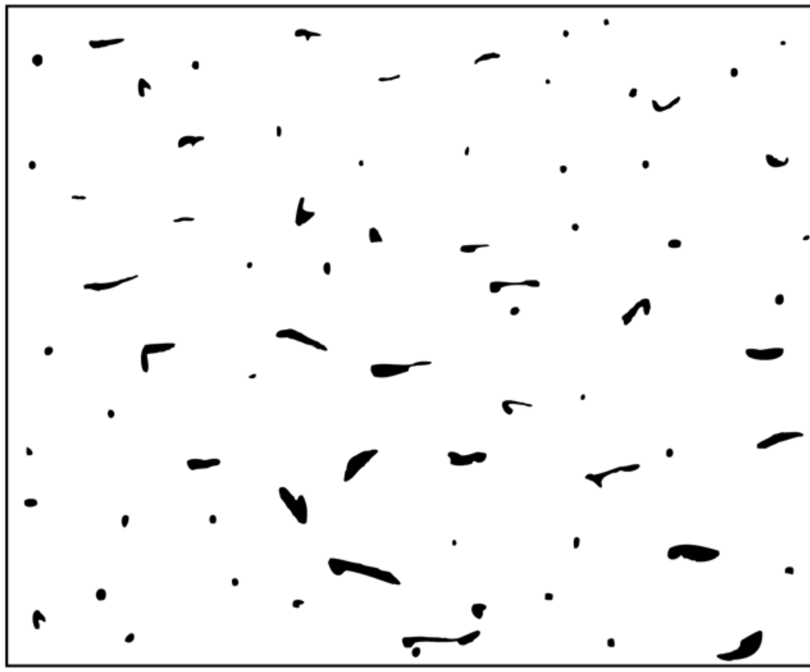


FIG. 3 — 2.0 % Porosity

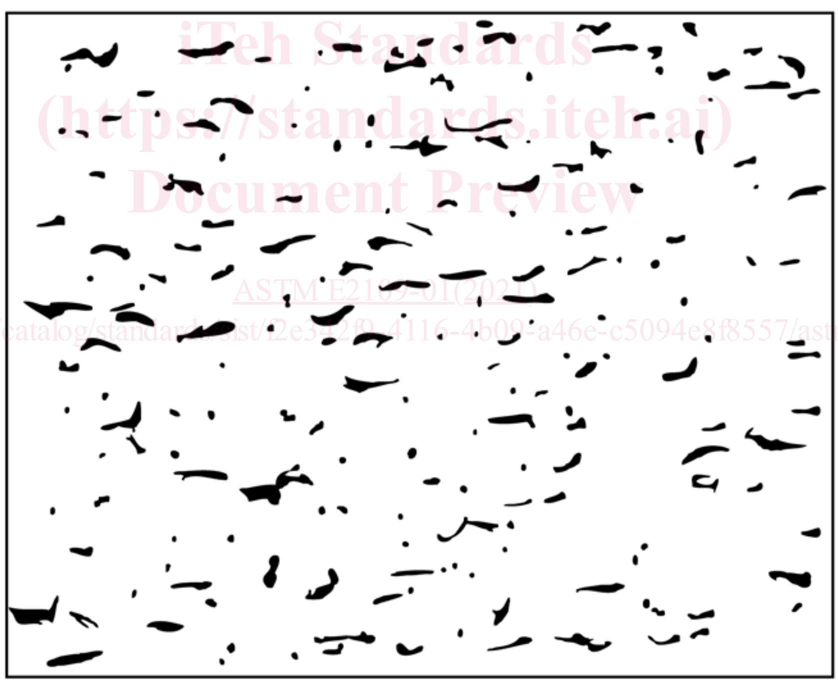


FIG. 4 — 5.0 % Porosity

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 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

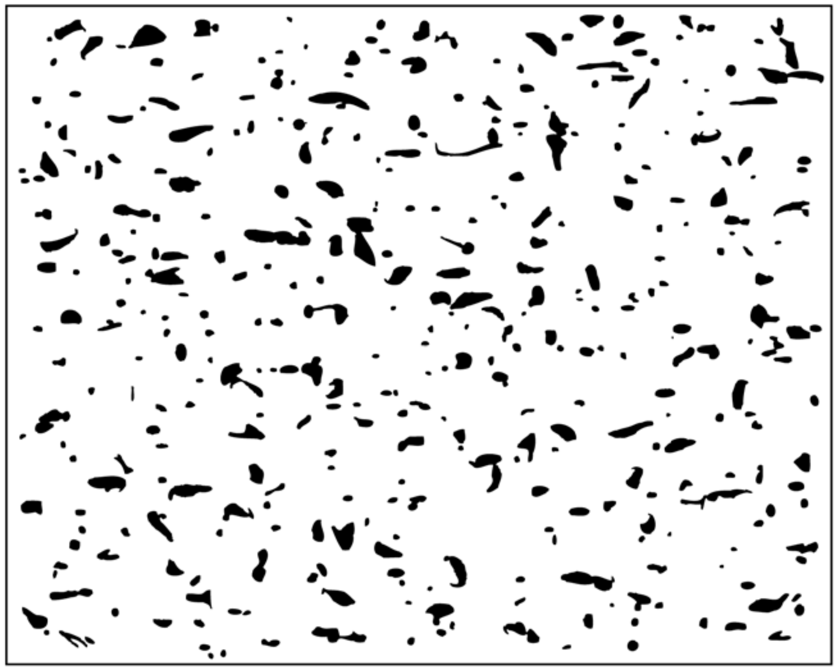


FIG. 5 — 8.0 % Porosity

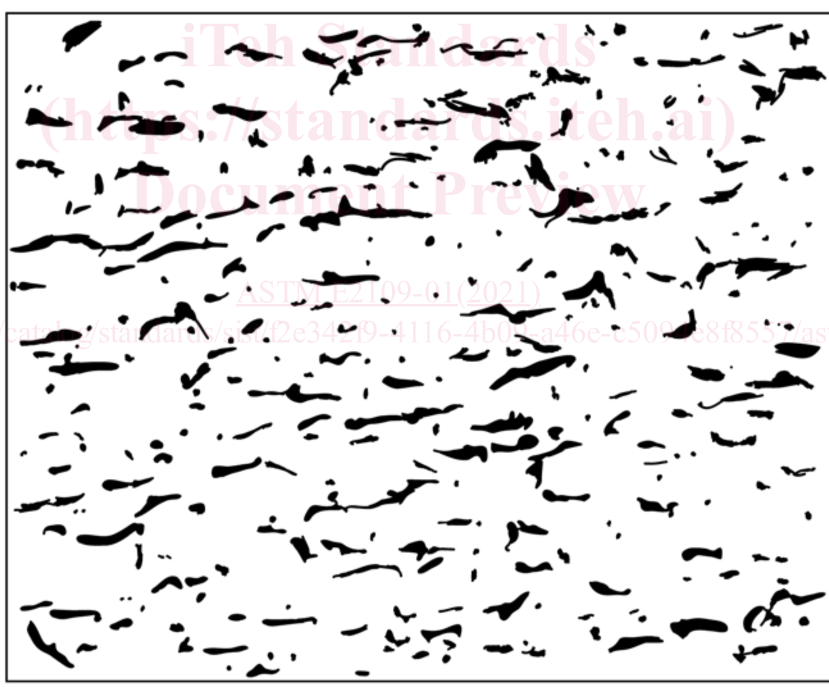


FIG. 6 — 10.0 % Porosity

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. 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

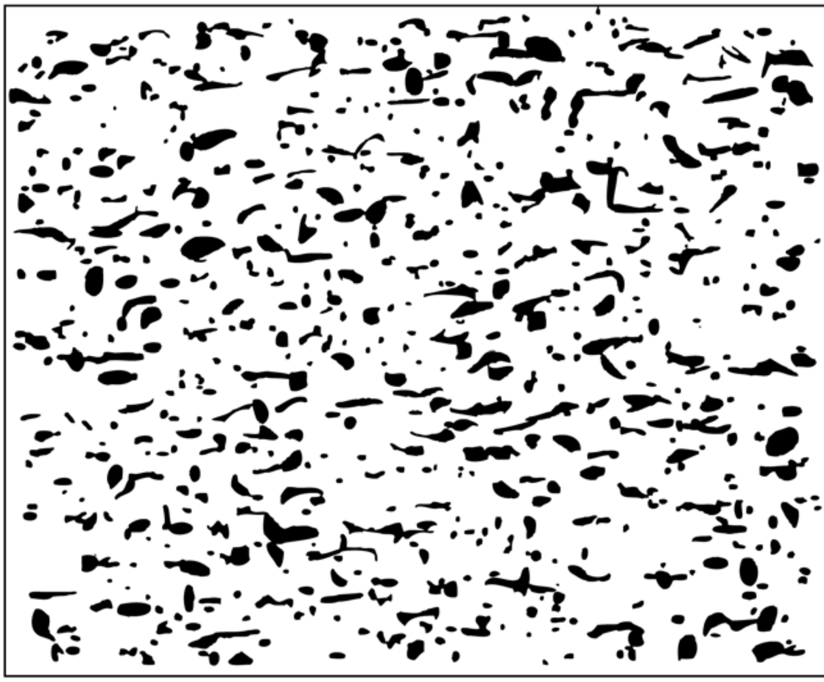
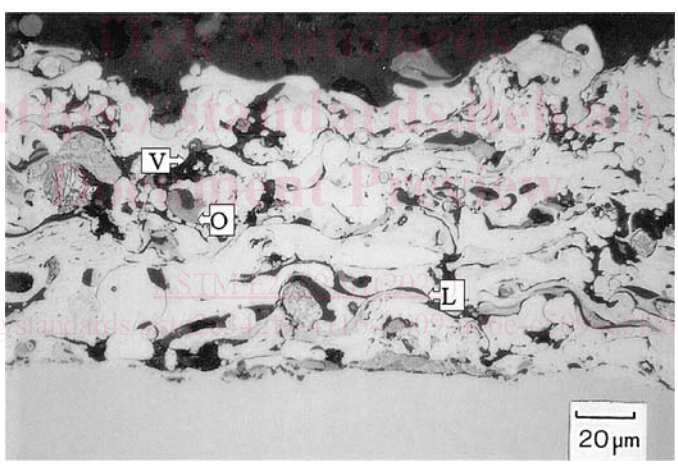


FIG. 7 — 15.0 % Porosity



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

FIG. 8 Ni/Al TSC—500X

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 and recommendations are given in Practice E3; 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 E1920.

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

[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.