Designation: C 1212 – 98 (Reapproved 2004)

Standard Practice for Fabricating Ceramic Reference Specimens Containing Seeded Voids[1]

This standard is issued under the fixed designation C 1212; 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 This practice describes procedures for fabricating both green and sintered test bars of silicon carbide and silicon nitride containing both internal and surface voids at prescribed locations.

1.2 The values stated in SI units are to be regarded as the standard.

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:* [2]

B 311 Test Method for Density Determination for Powder Metallurgy (P/M) Materials Containing Less than Two Percent Porosity

C 373 Test Method for Water Absorption, Bulk Density, Apparent Porosity, and Apparent Specific Gravity of Fired Whiteware Products

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *green specimen*—a ceramic specimen formed as originally compacted prior to high-temperature densification.

3.1.2 *internal void*—a cavity in a specimen with no connection to the external surface.

3.1.3 *seeded voids*—intentionally placed discontinuities at prescribed locations in reference specimens.

3.1.4 *sintered specimen*—formed ceramic specimen after firing to densify and remove solvents or binders.

3.1.5 *surface void*—a pit or cavity connected to the external surface of a specimen.

4. Significance and Use

4.1 This practice describes a method of fabricating known discontinuities in a ceramic specimen. Such specimens are needed and used in nondestructive examination to demonstrate sensitivity and resolution and to assist in establishing proper examination parameters.

5. Apparatus

5.1 *Aeroduster*, moisture-free.

5.2 *Die*, capable of exerting a pressure of up to 120 MPa, that will not contaminate the compacted material.

5.3 *Optical Magnifier*, capable of providing 10 to 30X magnification.

5.4 *Tubing*, latex, thin-wall, capable of withstanding isopress.

5.5 *Carver Press* or similiar type of appartus capable of exerting the necessary pressure to consolidate the sample.

5.6 *Cold Isostatic Press*, capable of maintaining 500 MPa.

5.7 *Vacuum Oven or Furnace* which can maintain a temperature of 525°C.

5.8 *Imaging Equipment* with the capability of producing a hard copy output of the image (that is, 35mm camera, CCD camera outputted to a video printer, a stereo microscope with 4 X 5 instamatic film, etc.).

5.9 *Sintering Furnaces* capable of reaching temperatures of 1400–2200°C. Depending on the ceramic system chosen, the furnace may be required to operate in a vacuum and/or under inert gas atmospheres at pressures as high as 200 MPa.

5.10 Commercial or similar device capable of measuring within .01 mg. Measuring densities according to Archimedes principle requires the use of a sample holder suspended in water attached to the scale.

6. Materials

6.1 *Silicon Carbide or Silicon Nitride Powders*, of appropriate purity and particle size, prepared with sintering aids and binder representative of the product to be inspected and in a manner appropriate for dry pressing with granule size less than 100-mesh.

[1] This practice is under the jurisdiction of ASTM Committee C28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.03 on Physical Properties and Non-Destructive Evaluation.

Current edition approved May 1, 2004. Published June 2004. Originally approved in 1992. Last previous edition approved in 1998 as C 1212–98.

[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.

6.2 *Styrene Divinyl Benzene Spheres*, with diameters as necessary. Other material with low vaporization temperatures may be substituted, but pressing characteristics and final void sizes may be different.

7. Fabrication of Surface Voids

7.1 *Green Specimens*:

7.1.1 The test piece geometry must be appropriate for the size and geometry limits of the NDE test method. If the purpose of the test is to determine if the NDE method is suitable for the detection of voids in a particular part/sample, ideally the test sample should be identitcal to the part/sample. If this is not feasible due to fabrication or testing limitations, the test sample should be similar to the part/sample in chemical composition, density, and thickness (the thickness of the test sample should be the same as the thickness in the area of the part/sample being examined.

7.1.2 *Procedure*:

7.1.2.1 Prepare the test specimen bars by pouring ceramic powder into a die in an amount sufficient to make a specimen of the desired thickness. Level the surface and press at a nominal pressure of 60 MPa.

7.1.2.2 Remove the ram to expose the specimen. Clean the specimen of all particles that are not flush with the top surface; this can generally be performed with a moisture-free aeroduster.

7.1.2.3 Place large spheres in the desired location on the specimen surface. Small microspheres may be moved to the desired position with a single human hair taped to a stiff plastic rod, using the assistance of an optical magnifier.

7.1.2.4 Press the spheres into the surface at a suitable pressure to obtain the desired strength for handling of the green compact (typically 120 MPa).

7.1.2.5 Remove the bar from the die and clear the surfaces of extraneous particles. An aeroduster or brush should be adequate.

7.1.2.6 Place the specimen in a thin-wall latex tube and evacuate the air. Seal the tube end. Cold isopress at a pressure suitable for a specific material (nominally 210–420 MPa).

7.1.2.7 Remove the specimen from the tubing and heat it in a vacuum to decompose the spheres (525°C for 45 min for styrene divinyl benzene).

7.1.2.8 Mark the specimen orientation with a scribe mark or by beveling a corner or edge. Remove extraneous particles from all surfaces with an aeroduster or brush. Light sanding may be necessary, for adherent particles.

7.1.3 *Void Measurement*—Measure the lateral dimensions in two orthogonal directions. The depth can be measured by focusing a microscope alternately on the specimen surface and on the bottom of the crater and noting the difference in the vertical position of the tube. Use a magnification suitable for measuring depth within 2 μm.

7.1.4 Measure the bulk density of the specimen from direct volume and weight measurements.

7.2 *Sintered Specimens*:

7.2.1 *Procedure*:

7.2.1.1 Follow the steps given in 7.1.2 to produce green specimens.

7.2.1.2 Sinter green samples under suitable conditions to achieve full densification. Nominal sintering conditions for silicon nitride are 1700–1900°C for 1 h in an inert atmosphere at 0–200 MPa; for silicon carbide, sintering temperatures of 2000–2200°C for 0.5 h under vacuum are commonly used. The sintering aids used will dictate the firing conditions. Measure the bulk density using either Test Method B 311 or Test Method C 373 or from volume and weight measurements.

7.2.2 *Void Measurement*—See 7.1.3.

7.3 *Surface Void Characteristics (for Both Green and Sintered Specimens)*:

7.3.1 Surface voids produced by this procedure are not spheroidal in shape. The final dimensions are a function of the compressibility of the seeded spheres and the compressibility and sintering characteristics of the powders that comprise the bulk material.

7.3.2 *Silicon Nitride Test Bars*—Made from 100-mesh powder containing yttria and silica sintering additives: The lateral surface dimensions of voids smaller than 100 μm are up to 10 % greater than the diameter of the seeded styrene divinyl benzene spheres. Surface dimensions of larger voids are approximately equal to the seeded sphere diameter. The depth-to-width ratio increases from 0.6 to 0.8 as the seeded sphere size increases from 50 to 115 μm.

7.3.3 *Silicon Carbide Test Bars*—Made from 100-mesh alpha silicon carbide powder; in green specimens, the lateral surface void dimensions are approximately 25 % greater than the diameter of seeded divinyl benzene spheres, while in sintered specimens they are approximately 10 % greater. The depth-to-width ratio is approximately 0.4 in both green and sintered specimens.

7.3.4 Compaction and burn-off usually cause powder particles to accumulate in the craters of green samples. If these are not removed prior to sintering (7.1.2.8) they will fuse to the walls, resulting in an irregular void with less volume.

8. Fabrication of Internal Voids

8.1 *Green Specimens*:

8.1.1 *Procedure*:

8.1.1.1 Prepare the test specimen bars by pouring powder into a die in an amount sufficient to position the voids at the desired distance from the specimen surfaces. If the voids to be seeded are smaller than 200 μm in diameter, level the surface and press at 60 MPa to facilitate positioning of the spheres. Pressing is not necessary if the diameter is greater.

8.1.1.2 To position spheres, follow 7.1.2.2 or 7.1.2.3, or both.

NOTE 1—If voids smaller than 100 μm are being seeded, it is advisable to insert discrete spheres at least 250 μm in diameter in selected locations to provide markers detectable with X rays.

8.1.1.3 Press the microspheres into the surface at a pressure of 60 MPa to hold them in position.

8.1.1.4 Record the positions of the spheres photographically with the use of imaging equipment or other suitable means.

8.1.1.5 Add ceramic powder in an amount sufficient to separate the adjacent layers of the voids. If this is the final layer of powder, press to provide handling strength to the green compact (nominally 120 MPa); otherwise, press at 60 MPa and

repeat the steps given in 8.1.1.2 through 8.1.1.4 until the desired number of void layers is obtained.

8.1.1.6 After final pressing, remove the specimen from the die and place it into thin-wall latex tubing, evacuate the air, and seal the end. Cold isopress at 420 MPa or a pressure most suitable for the specific material.

8.1.1.7 Remove the specimen from the tubing and heat it in a vacuum at 525°C for 45 min. to decompose the styrene divinyl benzene spheres.

8.1.1.8 Follow the procedure described in 7.1.2.8.

8.1.2 *Void Measurement*—To estimate the total void volume, remove extraneous particles from the specimen surfaces by sanding or brushing, and measure bulk density. Control specimens without seeded voids should be fabricated for comparison. Internal void dimensions can be inferred from direct measurements on selected samples by sanding off surface layers to expose the cavities. Alternatively, specimens can be broken so that void dimensions can be measured on both fracture surfaces.

8.2 *Sintered Specimens*:

8.2.1 *Procedure*:

8.2.1.1 Follow 8.1.1.

8.2.1.2 Follow 7.2.1.2.

8.2.2 *Void Measurement*—To estimate the total void volume, sand off any bumps that may cling to the surfaces and measure the bulk density. Control specimens without seeded voids should be fabricated for comparison. Internal void dimensions can be inferred from direct measurements on selected samples by grinding and polishing off surface layers to expose the cavities.

8.3 *Internal Void Characteristics (for Both Green and Sintered Specimens)*:

8.3.1 Characterization is complicated by the following factors:

8.3.1.1 Due to initial compaction by unidirectional pressing, the shape of the cavities is that of an oblate ellipsoid. The walls are not smooth-sided and thus resemble natural fabrication-induced voids.

8.3.1.2 During decomposition of the seeded spheres, some of the surrounding powder may be drawn into the cavity, resulting in a partially filled void.

8.3.2 Sintering reduces the size of the voids as well as that of the bar as a whole. The following[3,4] observations were made on specimens made from 100-mesh silicon nitride powder containing yttria and silica sintering aids and from 100-mesh alpha silicon carbide powder. The minor axis of voids seeded with 80-μm spheres was reduced by as much as 75 % in silicon nitride and 35 % in silicon carbide. The shrinkage of larger seeded voids was less. In the case of 200-μm seeded spheres, the reduction in the minor axis was approximately 35 % in silicon nitride and 20 % in silicon carbide.

9. Reporting Voids

9.1 Report the location of the voids. (For surface voids, use photographic methods.)

10. Precision and Bias

10.1 Insufficient data exist to establish consensus measured values for precision and bias.

11. Keywords

11.1 advanced ceramics; reference specimen; seeded voids; voids

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[3] Roth, D. J., Klima, S. J., Kiser, J. D., and Baaklini, G. Y., "Reliability of Void Detection in Structural Ceramics by Use of Scanning Laser Acoustic Microscopy," *Materials Evaluation*, Vol 44, No. 6, May, 1986, pp. 762–769. NASA TM 87035, 1985.

[4] Baaklini, G. Y., Kiser, J. D., and Roth, D. J., "Radiographic Detectability Limits for Seeded Voids in Sintered Silicon Carbide and Silicon Nitride," *Advanced Ceramic Materials*, Vol 1, No. 1, 1986. NASA TM 86945, 1984.