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Standard Practice for Fabricating Non-Oxide Ceramic Reference Specimens Containing Seeded Inclusions[1]

This standard is issued under the fixed designation C1336; 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 covers a procedure for fabricating both green and sintered test samples of silicon carbide and silicon nitride containing inclusions. These samples can be used to determine the sensitivity and detection capability of a nondestructive examination (NDE) method.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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]

B331 Test Method for Compressibility of Metal Powders in Uniaxial Compaction

C373 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*—formed ceramic specimens as originally compacted prior to high-temperature densification.

3.1.2 *inclusion*—a solid discontinuity in a specimen, with a composition not that of the specimen.

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

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

4. Significance and Use

4.1 This practice describes a method of fabrication of 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 *Aerosol Duster,* moisture-free.

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

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

5.4 *Tubing,* latex, thin-wall, for encapsulating compact during isopressing.

5.5 *Carver Press,* or similar type of apparatus capable of exerting the necessary pressure to consolidate the sample.

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

5.7 *Oven or Furnace,* which can maintain a temperature of 500°C.

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

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

5.10 *Mettler Scale,* or similar device capable of measuring within 0.01 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

[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.

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[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.

binder representative of the product to be inspected and in a manner appropriate for dry pressing with granule size less than −100 mesh.

6.2 *Inclusion Spheres,* diameters as needed. Inclusion materials selected based on applicable green and sintered inclusion that is desired to be investigated for a specific silicon nitride or silicon carbide application.

6.2.1 The size, shape, and composition of the inclusions shall be determined a priori before seeding. The inclusion particles must be carefully selected based on the ceramic system of interest and the purpose of the test samples.

6.2.1.1 Samples fabricated to determine the sensitivity and resolution of the NDE test method should have inclusions that represent the type that would be encountered in actual applications. The selection of the inclusion type should reflect the possible impurities that are commonly seen after processing and that are present in the raw material.

6.2.1.2 Test samples being fabricated for NDE calibration standards may require the use of inclusion particles with well-defined properties (that is, geometry, density, modulus, composition, etc.). Such particles may be chosen to be exceptionally stable during sample fabrication.

7. Fabrication of Internal Seeded Inclusions

7.1 *For 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 inclusions in a particular part/sample, ideally the test sample should be identical 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 test specimen bars by pouring powder into a die in an amount to position the inclusions at the desired distance from the specimen surfaces. If the inclusions to be seeded are less than 200 µm in diameter, level the surface and press at 60 MPa to facilitate positioning of the inclusions. Pressing is not necessary if the diameter will be greater.

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 done with a moisture-free aerosol duster.

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

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

7.1.2.5 Record the positions of the inclusions photographically using a CCD camera attached to a stereo microscope or other suitable imaging equipment depending on the size of the inclusions used.

7.1.2.6 Add ceramic powder of a sufficient amount to separate the adjacent layers of inclusions. 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 7.1.2.2 – 7.1.2.5 through until the desired number of inclusion layers are obtained.

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

7.1.2.8 Remove the specimen from the tubing and heat to a sufficient temperature to decompose the binder if it has been added for powder compaction assistance.

7.1.2.9 Mark the specimen orientation with a scribe mark or by beveling a corner or edge. Remove extraneous particles from all surfaces with an aerosol duster or brush (adherent particles may require light sanding).

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

7.2 *For Sintered Specimens:*

7.2.1 *Procedure:*

7.2.1.1 Follow steps 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 to 1900°C for 1 h in an inert atmosphere at 0 to 200 MPa; for silicon carbide, sintering temperatures of 2000 to 2200°C for 0.5 h under vacuum are commonly used. The sintering aids used will dictate the firing conditions. Measure bulk density using a method referenced in 2.1 (Test Methods B331 and C373) or from volume and weight measurements.

7.3 *Sintered Inclusion Characteristics:*

7.3.1 *For Silicon Nitride Test Bars*—Many inclusions react with silicon nitride powder and sintering aid powders during densification, potentially resulting in chemical and structural changes to the inclusion and possibly resulting in a reaction zone different in size from the size of the original inclusion before densification. For example, iron inclusions can react with free silica in the silicon nitride powder during densification to form iron silicide, with a larger resulting reaction zone than the original iron inclusion size. The dimensions of the inclusion may increase by 50 %. The final dimensions of the inclusion are affected by the environmental conditions within the furnace, cooling rates, soak times, etc. Thus, the effect densification has on inclusions in silicon nitride should be known a priori to ensure correct interpretation of results.

7.3.2 *For Silicon Carbide Test Bars*—Many inclusions may also react with the SiC powder and some of the sintering aid powders during densification. This reaction may result in structural and chemical changes to the inclusion causing the dimensions of the inclusion to be different than that of the original. The reactivity of the inclusion with the SiC powders or additives should be understood for proper interpretation of the results.

8. Fabrication of Surface Seeded Inclusions

8.1 *For Green Specimens:*

8.1.1 *Procedure:*

8.1.1.1 Prepare test specimen bars by pouring 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.

8.1.1.2 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 done with a moisture-free aerosol duster.

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

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

8.1.1.5 Record the position of the inclusions with the use of imaging equipment or other suitable means.

8.1.1.6 Remove the bar from the die and clear surfaces of extraneous particles. An aerosol duster or brush should be adequate.

8.1.1.7 Remove the specimen from the die and place into thin-wall latex tubing, evacuate the air, and seal the end. Cold isopress at 420 MPa or a pressure most suitable for specific material.

8.1.1.8 Remove the specimen from the tubing and heat to a sufficient temperature to decompose the binder if it has been added for powder compaction assistance.

8.1.1.9 Mark the specimen orientation with a scribe mark or by beveling a corner or edge. Remove extraneous particles from all surfaces with an aerosol duster or brush (adherent particles may require light sanding).

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

8.2 *For Sintered Specimens:*

8.2.1 *Procedure:*

8.2.1.1 Follow steps in 8.1.1 to produce green specimens.

8.2.1.2 Sinter green samples under suitable conditions to achieve full densification. Nominal sintering conditions for silicon nitride are: 1700 to 1900°C for 1 h in an inert atmosphere at 0 to 200 MPa; for silicon carbide, 2000 to 2200°C for 0.5 h under vacuum is commonly used. The sintering aids used will dictate the firing conditions. Measure bulk density using a method referenced in 2.1 or from volume and weight measurements.

9. Report

9.1 Report the location and measured size of the inclusion. Depending on the size of the inclusion and the precision required, the inclusion can be measured directly or by indirect methods using suitable imaging equipment.

10. Keywords

10.1 advanced ceramics; non-oxide ceramics