National Institute of Standards & Technology

Certificate

Standard Reference Material® 2100a

Fracture Toughness of Ceramics

This Standard Reference Material (SRM) is intended for verification of fracture toughness testing procedures. This SRM may be used with any fracture toughness test method, but is optimized for beam bending testing configurations. A unit of SRM 2100a consists of a set of five hot-pressed silicon nitride flexure specimens cut from a single billet (plate) of material.

The SRM may be used in conjunction with American Society of Testing and Materials (ASTM) fracture toughness standard C1421-99 [1]. This SRM may also be used with three International Standards Organization (ISO) standard tests prepared by ISO Technical Committee TC 206, Fine Ceramics [2,3,4].

Certified Fracture Toughness: A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [5]. NIST tested a large number (15 % to 20 % of the total) of specimens taken at random from three billets (see Appendix A, Specimen and Crack Plane Orientation). Most of the specimens were tested at NIST by the surface crack in flexure (SCF) and precracked beam [PB, also known as single-edged precracked beam (SEPB)] methods in accordance with ASTM C1421 [1], see Figure 1. In addition, ten specimens taken from SRM 2100 (billet C), one of the three billets, were also tested by the chevron notch in bending (CNB) test configuration A, also in accordance with C1421 [1]. The mean toughness values as measured by the three test methods agreed to within 0.4 % to 1.6 %, depending upon the billet, confirming that fracture toughness was independent of test method. Additional details are given in Appendix B.

Table 1. Certified Fracture Toughness[(a)]

Billet	Fracture Toughness, K_{Ic}	Uncertainty, U_1, in Fracture Toughness of a Single Specimen	Uncertainty, U_m, in the Mean Fracture Toughness For n = 5 Specimens
G	4.268 MPa·m^{½}	0.307 MPa·m^{½} (7.2 %)	0.145 MPa·m^{½} (3.4 %)

(a) The uncertainties are at the 95 % confidence level. The numbers in parenthesis are the uncertainties expressed as a percentage of the mean fracture toughness. The measurand is the mean fracture toughness listed in Table 1. Metrological traceability is to the SI derived unit for toughness (expressed as MPa·m^{½}).

The average fracture toughness and the scatter in results as measured by the three test methods were statistically indistinguishable, and the data, therefore, was pooled. The certified average fracture toughness in Table 1 is the grand average of the pooled NIST data base for billet G.

Expiration of Certification: The certification of **SRM 2100a** is valid indefinitely, within the measurement uncertainty specified, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Storage, Handling, and Use"). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

This SRM was prepared by G.D. Quinn of the NIST Materials Measurement Science Division, with assistance from K. Xu and R.J. Gettings formerly of NIST.

John A. Small, Chief
Materials Measurement Science Division

Gaithersburg, MD 20899
Certificate Issue Date: 03 March 2016

Steven J. Choquette, Acting Director
Office of Reference Materials

Statistical consultation was provided by N.F. Zhang of the NIST Statistical Engineering Division.

J.J. Swab of the U.S. Army Research Laboratory assisted with preliminary experiments. CNB tests were performed by J.A. Salem of NASA-Glenn Center.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

SOURCE, PREPARATION, AND ANALYSIS(1)

Description of SRM: This SRM was prepared from material acquired during the 1970 to 1980's and consists of a set of 5 unprecracked ceramic test specimens of hot-pressed silicon nitride (HPSN), Norton Grade NC 132, manufactured by Norton Co. (Worcester, MA). The NC 132 HPSN was originally fabricated in the form of a billet of nominal size 158 mm × 158 mm square with a thickness of 20 mm to 30 mm. The specimens were sliced from the billet such that the 3 mm × 4 mm cross section plane (in which cracks will be initiated and propagated) is parallel to the hot pressing direction as described in detail in Appendix A. This material was hot-pressed with a small amount (about 1 wt %) of magnesium oxide (or a magnesium compound which yields MgO when heated) sintering aid and is primarily composed of slightly elongated beta silicon nitride grains. A small amount of amorphous second phase resides in small triple point pockets or as thin layers between the silicon nitride grains. Some small tungsten carbide inclusions are dispersed throughout the material. This material fractures in a mixed transgranular and intergranular mode. It has a flat R-curve(2) and is highly resistant to environmentally-assisted slow crack growth at room temperature. Consequently, different test methods produce virtually identical toughness results.

There is a statistically significant difference in fracture toughness for specimens from different billets. There were three separate billets produced from the acquired material. The specimens in this SRM kit are all from a single billet designed "G" which is listed in the first column of Table 1 and scribed onto one end face of each specimen.

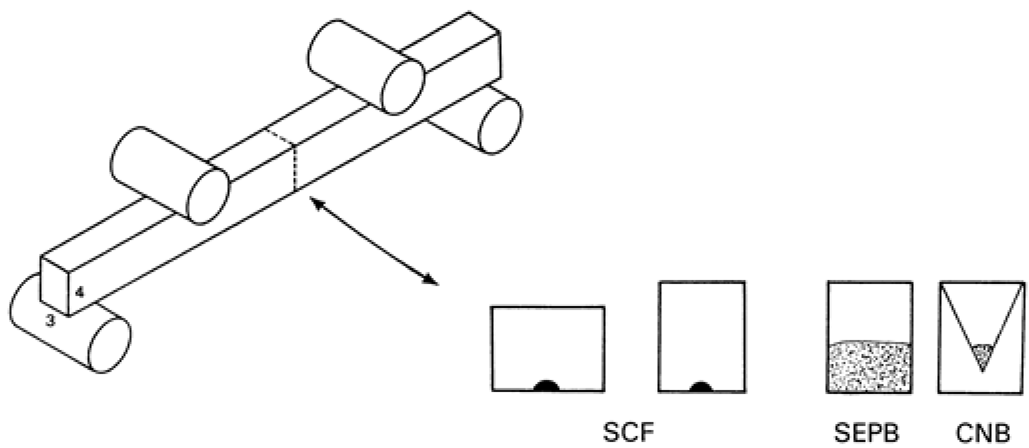


Figure 1. The three fracture toughness test methods in C1421 and in ISO 18756, ISO 15732, and ISO 24370. The insert depicts the specimen cross sections and the types of precracks. Note that the SCF specimens may be precracked and tested in either of two possible orientations.

Each 3 mm × 4 mm × (45 to 47) mm flexure bar specimen was prepared in accordance with conventional flexure strength standards MIL STD 1942(A) [6], ASTM C 1161 [7], and ISO standard 14704 [8], as well as with ASTM C1421 [1] and ISO 18756 [2], ISO 15732 [3], and ISO 24370 [4] for fracture toughness testing of ceramics. In addition, one 4 mm wide face has a 900 grit finish which is finer than is normally required and is provided as a convenience to users who wish to precrack with a Knoop indenter for the SCF test method. The edges of the specimens have been chamfered in accordance with requirements for the surface crack in flexure (SCF) and single-edged precracked beam (SEPB) test methods. (A small correction factor for the chamfer should be applied to SCF results as described in Appendix C.)

(1) Certain commercial equipment, instruments or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

(2) Crack growth resistance (R) is independent of crack size.

Discussion of Uncertainties: The uncertainty estimates were made according to the ISO/JCGM Guide [9] and consisted of Type A uncertainties evaluated by statistical means. NIST tested a large number (15 % to 20 % of the total) of specimens taken at random from each billet used. Most of the specimens were tested to fracture by one of the three standard test method procedures: SCF, CNB, or PB (SEPB). Additional PB (SEPB) experiments were performed on the broken halves.

The uncertainty, U_1, in Table 1 is a 95 % prediction uncertainty for a single future observation, based on the results of the NIST independently and randomly selected observations from the same population [10]. The uncertainty, U_m, is a 95 % prediction uncertainty for the mean of five future observations, also based on the results of the NIST independently and randomly selected observations. The expanded uncertainty is calculated as $U = ku_c$ where u_c is, at the level of one standard deviation, and represents the combined effects of material inhomogeneity and within-laboratory components of uncertainty. U_1 is the uncertainty for an individual specimen, and U_m is the uncertainty in the mean fracture toughness for the five specimens in this kit. The coverage factor, k, is determined from the Student's t-distribution corresponding to the degrees of freedom appropriate to the number of specimens tested at NIST and at the 95 % confidence level.

INSTRUCTIONS FOR STORAGE, HANDLING AND USE

Storage and Handling: The specimens are packaged in conventional ceramic flexure specimen trays. The specimens are durable; no special handling requirements are necessary. Once precracked, the CNB and SEPB specimens should be handled with care to avoid accidental breakage.

Use: This SRM may be used with any fracture toughness test method although the specimens have been optimized for flexural modes of loading. The user must precrack the specimens. It is strongly recommended that the user test these specimens only after they have acquired some familiarity with the test method.

The certified fracture toughness values are valid for cracks propagated in planes that are parallel to the specimen 3 mm × 4 mm cross section. Cracks may be initiated or propagated from either the 3 mm wide or the 4 mm wide surfaces. The fracture toughness is not certified for cracks propagated on planes parallel to the specimens' long axis. See Appendix A for further details.

Consult ASTM C1421 [1] or ISO 18756 [2], ISO 15732 [3], or ISO 24370 [4] for specific test method details if the SCF, PB (SEPB), or CNB methods are used.

Test Specimens: Although the specimens are from a single billet, their dimensions should be measured individually due to slight variations in machining. The specimen chamfers should also be measured if the SCF method is used.

Test Fixtures: The specimens are suitable for flexural modes of loading with conventional flexure strength fixtures. Common 20 mm × 40 mm (or 10 mm × 20 mm) four-point flexure strength fixtures that are designed to be in accordance with the well-known standard flexure strength test standards are satisfactory for all three fracture toughness methods. Users are reminded that there are critical requirements for fixture alignment, load pin and specimen alignment, fixture articulation, and load pin rolling action to eliminate friction constraints that will bias the test result.

Four-point flexural loading is preferred for SCF and CNB testing. Spans of 20 mm × 40 mm are recommended, but not required. The PB (SEPB) test may be conducted in either three- or four-point loading. The outer span should be between 16 mm and 40 mm. Three-point loading should be performed with extra caution since it may be difficult to align the precrack with the middle roller, especially with short 16 mm to 20 mm spans.(3) The specimens in the SRM kit may be fractured on full-sized 20 mm × 40 mm fixtures. The halves may then be precracked and retested on shorter three- or four-point fixture spans.

Testing Rates: Testing rates between 0.1 $MPa \cdot m^{1/2}/s$ and 2.75 $MPa \cdot m^{1/2}/s$ are specified for normal testing [1]. Fracture toughness is independent of test environment (in laboratory air ambient conditions, 20 °C to 25 °C, with relative humidities of 20 % to 60 % or in dry-nitrogen gas) when normal testing rates were employed.

(3) Some tests are apt to be unacceptable and rejected due to twisted fracture planes.

SCF Precracking and Testing: The following information may be helpful if the SCF method is utilized. SCF specimens may be precracked at a 24.5 N (2.5 kgf) or 49.0 N (5 kgf) Knoop indentation load. The 24.5 N load is preferred since the resulting indentation is smaller, and less material needs to be removed by polishing or hand grinding after indentation. Most of the NIST SRM database was generated with this load, and a VAMAS round robin confirmed that this load was eminently suitable. One 4 mm wide face has been given a finer finish (900 grit) than is customary for flexure specimens and is suitable for Knoop indentation. The Knoop indentation alternatively may be implanted on one of the 3 mm wide faces, but these should be fine ground, lapped, or polished to provide a flat, even surface for the Knoop indentation.(4) If an indentation load of 24.5 N is used and the proper amount of material has been removed by polishing or hand grinding with abrasives, the precrack size should be approximately 50 µm deep × 150 µm wide. Machining the residual stress damage zone off the specimen's surface with the use of diamond grinding wheels is possible, but not recommended.(5)

Four-point loading is required for the SCF configuration since it is very difficult to align the tiny precrack with the middle load roller in three-point loading. Fracture loads are modest, (typically 400 N to 600 N for 24.5 N precracks in 20 mm × 40 mm four-point fixtures), so no special handling precautions need be taken. The flexure strength of the flawed specimen, and thus its fracture toughness, should be corrected for the chamfers in SCF tests. An average chamfer size should be measured and used for the correction as described in Appendix C. Consult ASTM C1421 for additional details. Appendix D has details on the precision error associated with this test method.

CNB Precracking and Testing: The specimens in this SRM are in accordance with configurations "A" and "D" in ASTM C1421, which require four-point loading. Proper chevron notch preparation is critical. The notch should be uniform, the grooves should align very closely, and the back sides of the chevron (dimensions a_{11} and a_{12} in ASTM C1421) must not cut through to the specimen back face. Causal notching with a diamond cut off wheel is usually unsatisfactory. A precision set up and specimen holder is optimal.

The "A" configuration CNB specimen-fixture combination in ASTM C1421 requires four-point flexure testing. Fracture loads are small, (typically 20 N to 50 N depending upon loading configuration), so care should be exercised in handling and preloading these specimens. Cracks should pop in or extend stably from the chevron tip during a normal fracture toughness test provided that the chevron notch has been prepared properly. The specimens in this SRM have been prepared with edge chamfers which are not required or recommended for CNB testing, but experience has shown that the chamfers do not interfere with the results for this SRM. Consult ASTM C1421 for additional details. Appendix D has details on the precision error associated with this test method.

PB (SEPB) Precracking and Testing: The following information may be helpful if the PB (SEPB) method is utilized. Conventional bridge precrackers may be used to pop in a precrack that is between 0.35 W and 0.60 W deep, where W, the specimen thickness, is 4 mm. A starter flaw is carefully centered in the bridge anvil precracker. A single 98 N (10 kgf) Knoop has been found to be satisfactory, provided that the long axis is lined up perpendicularly to the specimen long axis. The indentation may be placed directly into the ground 3 mm wide face. A finely ground or polished surface is unnecessary. With a 6 mm bridge gap, bridge anvil pop-in loads should be between 8 000 N and 11 000 N. One or more aligned Vickers indentations may be used, provided that the indentation load does not exceed 98 N (10 kgf). A shallow saw cut may also serve as a starter flaw.

Either three-or four-point loading may be used, but extra precautions are necessary with the former to guarantee that the precrack is lined up with the middle roller. Fracture loads will be small (50 N to 120 N depending upon testing configuration and precrack size), so care should be exercised in handling and preloading the specimens. No correction for specimen chamfers should be made. Consult ASTM C1421 for additional details. Appendix D has details on the precision error associated with this test method.

Other Precracking Testing Methods: Other test methods may be used with this SRM, but since the specimens are optimal for flexure testing, some configurations such as double torsion, double cantilever beam, etc., are not feasible. The Vickers indentation crack length method is not recommended due to the lack of a universally accepted equation for relating crack length to toughness, the lack of a universally accepted indentation load, and other experimental difficulties which have been confirmed by a VAMAS round robin project on similar materials [11]. Crack plane orientation is also an issue with this method, since it is known that the fracture toughness is different for cracks extending in the plane perpendicular to the hot-pressing direction compared to cracks running parallel to the

(4)One method utilized at NIST is to use a motorized hobby drill (e.g. Dremel) with a cloth polishing wheel and diamond paste to polish a small spot in the middle of the specimen.

(5) Experiments at NIST confirmed that the identical mean fracture toughness is obtained, but the standard deviation was considerably larger. The SCF precracks were not as regular as those from hand ground or lapped specimens. Apparently the machine grinding altered the precracks and possibly propagated portions of the crack front. It is also difficult to guarantee that each specimen has had the correct amount of material removed when using diamond wheel grinding, especially if the specimens are mounted and ground together.

hot-pressing direction. Under no circumstances should this SRM be used to generate a so-called "universal calibration constant" for the Vickers indentation crack length method, since it is impossible for a single constant to apply to a wide range of ceramics.

Repeat Testing: Broken halves of the specimens may be retested. PB (SEPB) testing at NIST on two billets demonstrated there was no difference in outcomes for full-size specimens tested on 20 mm × 40 mm four point fixtures versus outcomes from specimen halves tested on 10 mm × 20 mm spans, or halves tested on three-point fixtures with 20 mm spans. The reject rate for the three point configuration was greater, however, due to crack twisting and alignment problems.

Additional Information: Additional information on the development of the three billets is provided in references 22 to 25. Reference 22 provides additional information on how the uncertainties were calculated.

REFERENCES

[1] ASTM C1421; Standard Test Methods for Fracture Toughness of Advanced Ceramics; *In Annu. Book ASTM Stand.*, Vol. 15.01; ASTM: West Conshohocken, PA.

[2] ISO 18756; Fine Ceramics (Advanced Ceramics, Advanced Technical Ceramics) – Determination of Fracture Toughness of Monolithic Ceramics at Room Temperature by the Surface Crack in Flexure (SCF) Method; *In Fine Ceramics*; ISO, TC 206.

[3] ISO 15732; Fine Ceramics (Advanced Ceramics, Advanced Technical Ceramics) – Test Method for Fracture Toughness of Monolithic Ceramics at Room Temperature by Single Edge Precracked Beam (SEPB) Method; *In Fine Ceramics*; ISO, TC 206.

[4] ISO 24370; Fine Ceramics (Advanced Ceramics, Advanced Technical Ceramics) – Test Method for Fracture Toughness of Monolithic Ceramics at Room Temperature by Chevron-Notch Beam (CNB) Method; *In Fine Ceramics*; ISO, TC 206.

[5] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at http://www.nist.gov/srm/upload/SP260-136.PDF (accessed Mar 2016).

[6] MIL STD 1942A; *Flexural Strength of High Performance Ceramics at Ambient Temperature*; U.S. Army Materials Technology Laboratory: Watertown, MA (8 November 1990).

[7] ASTM C1161; Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature; *In Annu. Book ASTM Stand.*, Vol. 15.01; ASTM: West Conshohocken, PA.

[8] ISO 14704; Fine Ceramics (Advanced Ceramics, Advanced Technical Ceramics) – Test Method for Flexural Strength of Monotonic Ceramics at Room Temperature; *In Fine Ceramics*; ISO, TC 206.

[9] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Mar 2016); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at http://www.nist.gov/pml/pubs/tn1297/index.cfm (accessed Mar 2016).

[10] Hahn, G; Meeker, W.; *Statistical Intervals*: *A Guide for Practitioners*; John Wiley and Sons: NY (1991).

[11] Quinn, G.D.; Salem, J.A.; Bar-on, I.; Cho, K.; Foley, M.W.; Fang, H.; *Fracture Toughness of Advanced Ceramics at Room Temperature*; J. Res. NIST, Vol. 97 (5), pp. 579–607 (1992).

[12] Petrovic, J.J.; Mendiratta, M.G.; Fracture from Controlled Surface Flaws; *In Fracture Mechanics Applied to Brittle Materials*, ASTM STP 678; Freiman, S., Ed.; ASTM: West Conshohocken, PA, pp. 83–102 (1979).

[13] Petrovic, J.J.; Dirks, R.A.; Jacobson, L.A.; Mendiratta, M.G.; *Effects of Residual Stresses on Fracture from Controlled Surface Flaws*; J. Am. Ceram. Soc., Vol. 59(3-4), pp. 177–178 (1976).

[14] Quinn, G.D.; Gettings, R.J.; Kübler, J.J.; *Fracture Toughness of by the Surface Crack in Flexure (SCF) Method: Results of the VAMAS Round Robin*; Ceram. Eng. and Sci. Proc., Vol. 15(5), pp. 846–855 (1994).

[15] Quinn, G.D.; Kübler, J.J.; Gettings, R.J.; *Fracture Toughness of Advanced Ceramics by the Surface Crack in Flexure (SCF) Method: A VAMAS Round Robin*; VAMAS Technical Report #17; NIST: Gaithersburg, MD (June 1994).

[16] Quinn, G.D.; Gettings, R.J.; Kübler, J.J.; Fracture Toughness of Ceramics by the Surface Crack in Flexure (SCF) Method; *In Fracture Mechanics of Ceramics*, Vol. 11; Bradt, R.C.; Hasselman, D.P.H.; Munz, D.; Sakai, M.; Yashevchenko, V., Eds.; Plenum, NY, pp. 203–218 (1996).

[17] Quinn, G.D.; Gettings, R J.; Kübler, J.J.; Fractography and the Surface Crack in Flexure (SCF) Method for Evaluating Fracture Toughness of Ceramics; *In Fractography of Glasses and Ceramics, Ceramic Transactions*, Vol. 64; ACS: Westerville, OH, pp. 107–144 (1996).

[18] Nose, T.; Fuji, T.; *Evaluation of Fracture Toughness for Ceramic Materials by a Single-Edge-Precracked-Beam Method*; J. Am. Ceram. Soc., Vol. 71(5), pp. 328–333 (1988).

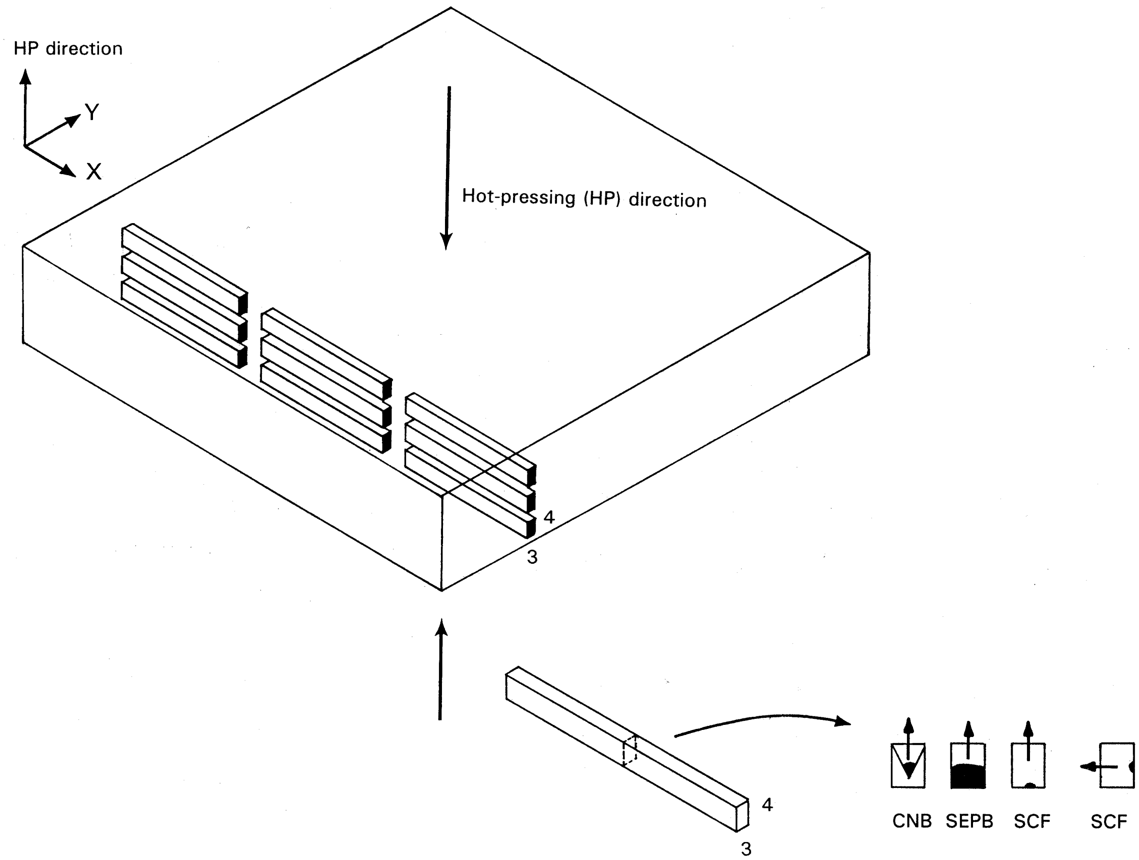
[19] Salem, J.; Shannon, J., Jr.; Jenkins, M.; Some Observations in Fracture Toughness and Fatigue Testing with Chevron-Notched Specimens; *In Chevron Notch Test Experience: Metals and Non-Metals*, ASTM STP 1172; Brown, K.; Baratta, F., Eds., ASTM: West Conshohocken, PA, pp. 9–25 (1992).
[20] Baratta, F.I.; Quinn, G.D.; Matthews, W.T.; *Errors Associated With Flexure Testing of Brittle Materials.*; Technical Report MTL TR 87-35; U.S. Army Materials Technology Laboratory: Watertown, MA (July 1987).
[21] ASTM E 691; *Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method*; Vol. 14.02, ASTM: West Conshohocken, PA.
[22] Quinn, G.D.; Xu, K.; Salem, J.A; Swab, J.J.; SRM 2100: The World's First Fracture Toughness Reference Material; *In Fracture Mechanics of Glasses and Ceramics*, Vol. 14; Bradt, R.C.; Munz, D.; Sakai, M.; White, K.W., Eds.; Klewer/Plenum: NY, pp. 499–530 (2005).
[23] Quinn, G.D.; Xu, K.; Gettings, R.; *Standard Reference Material 2100: Fracture Toughness of Ceramics*; Ceram. Eng. and Sci. Proc.; Vol. 20(3), pp. 513–523 (1999).
[24] Quinn, G.D.; Xu, K.; Gettings, R.J.; Salem, J.A.; Swab, J.J.; Standard Reference Material 2100: Ceramic Fracture Toughness; *In Fatigue and Fracture Mechanics*, Vol. 32, ASTM STP 1406; Chona, R., Ed., ASTM: West Conshohocken, PA, pp. 336–350 (2001).
[25] Quinn, G.D.; Xu, K.; Gettings, R.J.; Salem, J.A.; Swab, J.J.; Does Anyone Know the Real Fracture Toughness? SRM 2100: The World's First Ceramic Fracture Toughness Reference Material; *In Fracture Resistance Testing of Monolithic and Composite Brittle Materials*, ASTM STP 1409; Salem, J.A.; Quinn, G.D.; Jenkins, M.G., Eds. ASTM: West Conshohocken, PA, pp. 76–93 (2002).

Users of this SRM should ensure that the Certificate in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

APPENDIX A

Specimen and Crack Plane Orientation

All specimens were cut from the hot-pressed silicon nitride plates as illustrated in the figure below. Fracture toughness was measured and is certified only for crack planes that are *parallel to the hot-pressing direction*. Crack propagation may be in *either* the direction parallel to or perpendicular to the hot-pressing direction.



The small arrows show the direction of crack extension.

Identical results are obtained in SCF experiments with artificial precracks implanted on the narrow 3 mm specimen surfaces as compared to specimens with cracks implanted on the 4 mm faces. In addition, PB (SEPB) and CNB experiments wherein the crack extended in the direction as the 4 mm edge produced results identical to the SCF experiments wherein the crack started on the 4 mm wide face and propagated into specimens parallel to the 3 mm face.

ASTM C1421 recommends that the crack plane and the crack extension direction be defined in relation to the plate. The identification scheme is a set of letters first indicating the crack plane, then a second set of letters which designate the *direction of* crack extension in that plane. The first letter which describes the *crack plane* is designated by the vector that is *normal* to the crack plane. By this nomenclature scheme, all crack planes and crack extensions in this SRM are “X-HP”, “X-Y”, “Y-HP”, or “Y-X”. Fracture toughness is <u>not</u> certified for crack planes that are *perpendicular* to the hot-pressing direction (“HP-X” or “HP-Y” fracture systems). The fracture toughness is smaller for these crack planes.

APPENDIX B

Additional Background Information

The fracture toughness data for this SRM was generated by the three test methods in the ASTM standard test method [1].

The SCF method, also known as the "controlled flaw" method, is a development of early work by Petrovic and colleagues [12,13]. This innovative method utilizes a Knoop indenter to create a micro, semielliptical surface precrack. It is essential that residual stresses from the indentation are eliminated by polishing or hand grinding the indentation away after indentation. The specimen is fractured in a conventional flexural strength test fixture. Fractographic techniques are used to detect and measure the precrack after specimen fracture. The name of this method has been changed to "surface crack in flexure" in order to avoid confusion with other so-called controlled flaw methods and to make it consistent with fracture mechanics conventions. Fracture toughness is computed from the stress (or maximum load) at fracture, the crack size, and stress intensity factor solutions. The latter factor should be individually calculated for each specimen precrack. Additional details are in the literature [14-17].

The PB (SEPB) method is widely accepted in the fracture mechanics and ceramics community in large part since it is a traditional fracture mechanics configuration with a good record for producing accurate and precise results. A variant on the older single-edged notched beam method, the PB (SEPB) method uses an innovative precracking procedure (compression bridge-anvil loading) to produce a sharp precrack [18]. A saw cut or indentation is placed in the narrow side of a flexure specimen. The specimens are placed into a compression-anvil precracking apparatus and loaded carefully until a precrack pops in. The specimen is fractured in a flexure fixture. Crack size is measured on the fracture surface. Fracture toughness is computed on the basis of the specimen and fixture dimensions, the load at fracture, and stress intensity factor formulations.

The CNB test entails loading a chevron-notched specimen to fracture in a flexure fixture. The crack initiates at the chevron tip and then propagates stabily through the chevron. Since generalized stress intensity factor solutions do not exist for CNB testing, it is necessary to specify very specific specimen and notch configurations. The 3 mm × 4 mm cross section sized specimens in this SRM are suitable for use as either configuration A or D in ASTM C1421. Stringent requirements are placed upon the specimen preparation since successful outcomes, which rely upon stable crack extension, depend upon having well-machined notches [19]. Stress intensity factor coefficients are very sensitive to the precise chevron geometries. Analytical errors of 10 % or more are possible if formulas for idealized chevron geometries are applied to casually-fabricated specimens. It is especially important that: the two notch grooves (one on either side of the specimen) meet very closely without an offset, the notch tip be well centered, the sides of the chevron be very symmetric about the specimen centerline, and the sides of the chevron should end on the specimen side and not break through to the back surface. Proper specimen preparation requires careful machining by an experienced laboratory or machine shop, but experience shows this can be quite routine. Casual notch preparation by a student with a cut off wheel in a laboratory will not suffice. Misalignments in the chevron will promote unstable crack propagation that will invalidate the test. Experienced machine shops usually have a custom specimen holder to position the specimen while the chevron is cut from one side. The specimen or holder is flipped and the chevron completed by cutting from the opposite side. The specimen is tested in conventional flexure fixtures and the crack must be propagated stability. Fracture toughness is computed from the maximum load during stable crack extension, the specimen and fixture dimensions, stress intensity factor coefficient solutions, and the precise chevron geometry for each specimen.

A Versailles Advanced Materials and Standards (VAMAS) round robin which featured the SCF test method on NC 132 hot-pressed silicon nitride obtained extraordinarily consistent results from 20 laboratories around the world. Fracture toughness results for NC 132 in the literature from many sources, by many different test methods for over 20 years have been unusually consistent. The crack planes were parallel to the hot-pressing direction in most of these studies. In particular, results by the chevron notch in bending, short bar chevron notch, surface crack in flexure, and single-edged precracked beam tests converged in the range of 4.5 MPa·m^{½} to 4.9 MPa·m^{½}. The user is referred to references 14 to 16 for more information.

APPENDIX C

Chamfer Correction Factors, SCF Method

The fracture toughness of SCF specimens should be corrected for the corner chamfers. The correction increases the calculated SCF fracture toughness outcomes slightly (usually 0.4 % to 0.9 %). The NIST SCF fracture toughness data used to prepare the certified value for this SRM was corrected. Do <u>not</u> correct PB (SEPB) or CNB test results for corner chamfers.

The chamfers in the SRM specimens are typically 0.10 mm to 0.15 mm in size, although some may be larger. The chamfer size, c, may be measured with a microscope having a traveling stage or from photographic analysis. All eight c dimensions for the four chamfers should be measured and an average value used for the correction.

The fracture toughness in SCF specimens may be calculated from the formula:

$$K_{Ic} = Y \sigma_f \, a^{1/2}$$

where Y is the appropriate stress intensity shape factor (maximum), σ_f is the maximum flexure stress in the specimen at fracture, and a is the crack depth. See the ASTM C1421 or ISO 18756 SCF standards for more details on the calculation of K_{Ic} by this method.

The maximum flexure stress may be calculated from simple beam theory, and it is common to assume that the cross section is a simple rectangle. The chamfers alter this geometry, however, and the second moment of inertia of the specimen cross section about the neutral axis is altered [20]. The true maximum flexure stress at fracture is calculated as follows:

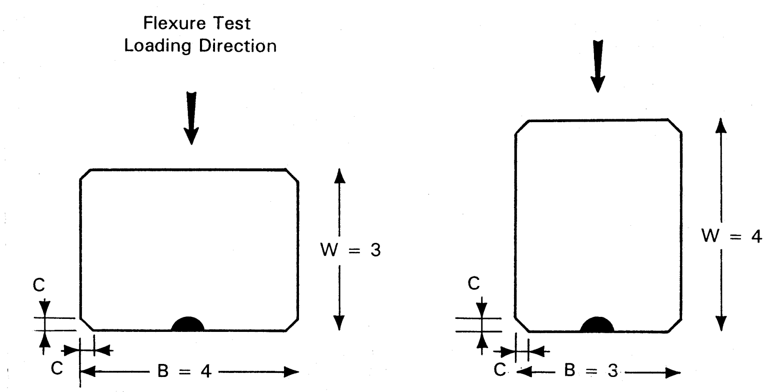
$$\sigma_f = F \, \sigma_b$$

where σ_b is the apparent flexure stress for a simple rectangular beam, computed from simple beam theory, and F is a correction factor. Correction factors are listed in the following tables and are practically identical for the two specimen orientations.

Fracture toughness then may be calculated:

$$K_{Ic} = Y\sigma_f \, a^{1/2} = Y\left(F\sigma_b\right)a^{1/2}$$

<u>Specimen Cross Section</u>



Correction Factor, F, for
<u>3 mm × 4 mm Specimens</u>

C (mm)	Correction factor, F B = 4, W = 3	Correction Factor, F B = 3, W = 4
0.080	1.003	1.003
0.090	1.004	1.004
0.100	1.005	1.005
0.110	1.006	1.006
0.120	1.007	1.007
0.130	1.008	1.008
0.140	1.009	1.009
0.150	1.011	1.011
0.160	1.012	1.012
0.170	1.014	1.014
0.180	1.015	1.015
0.190	1.017	1.017
0.200	1.019	1.019
0.210	1.020	1.021
0.220	1.022	1.023

APPENDIX D

Test Method Uncertainties and VAMAS Round Robins

The precision uncertainty for a measured value of fracture toughness depends upon the test method. The following information was not used in the preparation of the uncertainty statement for this SRM, but is included for the user's benefit to help the user appreciate the common sources of uncertainty.

SCF METHOD

Uncertainty of Individual Test Results by the SCF Method: In ASTM C1421, for the SCF test method, the type B uncertainties of the stress at fracture are estimated to be a maximum of 2 % to 3 %, the crack size measurement, 5 %, and the stress intensity factor coefficient, 3 % to 5 %. The latter two uncertainties are coupled, and it is estimated that this total uncertainty for fracture toughness is within 5 % [14-17].

A Versailles Advanced Materials and Standards (VAMAS) interlaboratory round robin [14-16] with 20 laboratories confirmed the practicality of the SCF test method and obtained estimates of the within-laboratory (repeatability) and between-laboratory (reproducibility) precision uncertainties. The type A estimates for repeatability and reproducibility are shown in Table 1. These results were obtained on the same material as used for this SRM (but from a different billet, designated "E"). These uncertainties are for an individual test specimen and include both the test method uncertainty and variability in the fracture toughness from specimen to specimen. See references 14 to 17 for further details.

Table 1. Precision for individual test results for the SCF method from a VAMAS round robin on NC 132 hot-pressed silicon nitride computed in accordance with ASTM Standard E 691 [21]. Data from 19 laboratories on specimens from a single billet "E". Results from one laboratory were outliers and were deleted.

			Repeatability (Within-Lab)			Reproducibility (Between-Labs)		
Average (MPa·m^{½})	Std. Dev[(a)] (MPa·m^{½})	Number of Specimens	Std. Dev. (MPa·m^{½})	95 % limit[(a)] (MPa·m^{½})	COV[(c)] (%)	Std. Dev. (MPa·m^{½})	95 % Limit[(b)] (MPa·m^{½})	COV[(c)] (%)
4.56	0.32	102	0.24	0.68	5.4	0.31	0.86	6.8

Notes:
(a) Standard deviation of all individual test results.
(b) Coverage factor of 2.8 in accordance with ASTM E 691 [21].
(c) Coefficient of variance expressed as a percentage of the average.

PB (SEPB) METHOD

Uncertainty of Individual Test Results by the SEPB Method: In ASTM C1421, the type B accuracy of the stress intensity factor coefficient for the SEPB test method is estimated to be a maximum of 2 %. No estimate was made for the total uncertainty.

ASTM C1421 cites results from a 16 laboratory international VAMAS round robin for a SEPB test procedure (similar to but not identical to that in C1421) on a gas pressure sintered silicon nitride.

CNB METHOD

Uncertainty of Individual Results by the CNB Method: In ASTM C1421, it is estimated that the type B accuracy of the stress intensity factor coefficient, Y_{min}^*, for the CNB test method is a maximum of 1 %.

No CNB round robin data are available at this time.