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Standardization of Fracture Toughness Testing of Ceramics in the United States

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American Society for Testing and Materials (ASTM) standard test method PS 070-97 has been created for measuring fracture toughness of advanced ceramics. PS 070-97 includes three test methods which use beams in bending: chevron notch (CNB), single-edged precracked beam (SEPB), and surface crack in flexure (SCF). Supporting data has been collected through several Versailles Advanced Materials and Standards round robins. This paper discusses the evolution of the standard including the rationale for the choice of the three methods and the specifications in the standard. Progress on Standard Reference material 2100 which will have certified values of fracture toughness is presented.

Key words : Fracture toughness, Standard, Ceramics, Referece material VAMAS

I. Introduction

The new ASTM provisional standard PS 070-97[1] features three specific test methods as shown in Fig. 1. Fracture toughness is a property of fundamental importance to ceramists and design engineers inasmuch as it is one measure of "brittleness". Other indices of brittleness are reviewed elsewhere.[2] Many methods have been devised to measure fracture toughness by both the fracture mechanics and ceramics communities who approach the matter with different perspectives.

The fracture mechanics community often adapt procedures developed for metals. These include compact tension, double-cantilever beam, chevron-notched beam, or straight-through notched beams in bending. The primary ASTM fracture toughness standard for metals, E 399,[3] is not particularly applicable to ceramics, however. Even the term K_{Ic} has been defined by the ASTM Committee E-08 "Frcture and Fatigue" in the context of metallic specimens. A critical issue for metals, whether the specimen is sufficiently large to be in plane strain versus plane stress loading, is not of concern with ceramics. Plastic zones, if they exist for ceramics, are so tiny that for all practical purposes, cracks experience only plane-strain conditions.

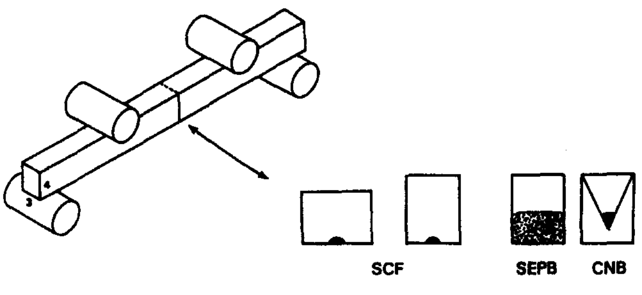

Fig. 1. The three test procedures in the ASTM fracture toughness standard. The SCF specimens may be tested in either orientation.

The ceramics community has devised or adapted new procedures such as the double torsion method and a variety of indentation methods. Single-edged notched beams (SENB) were popular with the ceramics community for many years, but gradually fell out of favor. It was recognized that even with the thinnest saw cuts, the notches were blunt and they were not true sharp precracks. This underscores one of the most fundamental requirements of a sound fracture toughness test: *the method should have a sharp, well characterized crack.* Precracking is one of the most critical elements of fracture toughness test methods.

Characterization of ceramic fracture toughness is complicated by two important phenomena: environmentally-ssisted subcritical crack extension ("slow crack growth" or SCG) and R-curve behavior. R-curve behavior refers to an increase in crack extension resistance as a crack propagates. For many materials the characterization of fracture toughness by a single value may be a gross oversimplification. R-curve phenomena also account for much contradictory fracture toughness test data published through the 1970's and 1980's. Eventually it was realized that large crack fracture mechanics specimens often produced results different than those from small crack test

[illegible]	**Brittle (no R-curve)** **SCG** K_I-V (Velocity) Curves
Rising R-Curve **No SCG** K_R - a Curves	**Rising R-Curve** **and SCG** K_R - a *and* K_I-V Curves

Fig. 2. Crack Growth/Fracture Response of Ceramics

specimens. A framework[a] illustrated in Fig. 2 helped characterize the fracture resistance of ceramics and was a key step in defining the scope of the standard. Materials with a flat R-curve could be considered "brittle" as contrasted with those materials with a rising R-curve. Materials also could be classed as to whether or not they experienced SCG. It was decided that the new standard should focus on materials characterized by the upper row, and in particular, the upper left cell: that is, materials that have negligible R-curve (no dependence of toughness on crack size) and negligible SCG. Nonetheless, the standard should incorporate provisions to at least detect SCG or R-curve behavior. Techniques to deal with SCG are well established, but are not well developed for R-curve behavior. We doubt whether a single test method can define an intrinsic R-curve since R-curves are specimen size, shape, stress state, and crack type specific.

ASTM attempts to standardize fracture toughness test methods began in the 1970's in Committee E-24, "Fracture Testing", a committee which traditionally had worked with fracture testing of metals. There was little progress in actually creating a ceramic standard test method, but the state of the art was well documented in five ASTM Special Technical Publications.4-8) Perhaps the most important was STP 6785) which summarized the state of the art of fracture toughness testing of ceramics in the late 1970's. The other STP's dealt with reliability evaluation,6) chevron-notch testing,7,8) and fractography.4)

In the meantime, a 1989 Versailles Advanced Materials and Standards (VAMAS) round robin project9,10) that featured the SEPB, indentation strength in bending (ISB), and indentation fracture (IF) methods organized by the Japan Fine Ceramic Center generated interest in standardization. The adoption of JIS R 160711) in February, 1990 with the SEPB method by the Japanese Standards Association and the creation of a draft standard DIN NMP 51 10912) in September, 1991 with both SENB and SEPB methods by the German Standards Institute also spurred fresh ASTM activity.

ASTM ceramic fracture toughness standardization had a resurgence in 1986 when committee C-28, Advanced Ceramics was founded. Fracture toughness was a high priority, but real progress did not commence until a workshop was held on "Fracture Toughness Testing of Ceramic Materials" at NASA's Lewis Research Center on December 4, 1991. This workshop was organized by Prof. Bar-On and Dr. J. Shannon of NASA-Lewis and was the starting point for the process that eventually led to the creation of PS 070-97. It was agreed that the project would be a joint undertaking of ASTM Committees C-28, "Advanced Ceramics" (which had primary jurisdiction), and E-08 "Fracture and Fatigue" (the successor to Committee E-24). A task group was formed comprising the four authors of this paper. Dr. E. Fuller of NIST and Mr. F. Baratta of the U. S. Army Research Laboratory also contributed.

The task group decided to make a single standard with a common core element and separate annexes for the various test methods (analogous to the structure of E 399). Although five methods initially were considered for incorporation in this early stage, surface crack in flexure (SCF), single-edged precracked beam (SEPB), chevron notch in bending (CNB), indentation strength in bending (ISB), and double-cantilever beam (DCB), attention eventually settled on the first three. The first draft was prepared by J. Salem in July, 1992. Subsequent drafts have been corrdinated by Profs. Bar-On and Jenkins. The first ASTM ballot was in April, 1995 and after additional balloting and revisions, the standard was adopted as provisional Standard, PS 070-97 in March 1997. An ASTM provisional standard is a document published for a limited time (2 years) to meet a demand for a more rapid issuance of specific documents. A provisional standard is adopted by a streamlined process and cannot be renewed. It is a useful means to get a test method "on the books" and to gain experience while a full-consensus version is refined and forged by the ASTM ballot process. Currently, a full-consensus version is in the final stages of development.

A few words about some of the alternative methods that were candidates for standardization are in order. The indentation strength in bending (ISB) procedure13) (in which a Vickers indenter creates cracks in a flexure specimen which is then fractured) was not pursued for many reasons. It was felt (particularly by the traditional fracture mechanics community) that the crack system was ill defined. Although test results often have low scatter, the mean test results often differ from those obtained from other methods. ISB fracture toughness often varies with indentation load and while this is commonly attributed to R-curve behavior, it also may be a purely geometric or stress gradient effect. For example, the stress intensity factor coefficients and crack shapes may change considerably as large semicircular or semi-elliptical surface cracks extend into small specimens loaded in flexure.14) The need to rely on a calibration constant (0.59±0.12)[b] that was determined by logarithmic regression analysis also led to unease. The original ISB paper

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[a] Suggested by E. Fuller, NIST.

[b] The uncertainty is assumed to be one standard deviation, but this is unclear from the original reference.

stated: "It should be possible to determine K_c for any well-behaved material to within 30~40% …".[13)] This assessment is quite candid, but this large uncertainty makes the results of limited value.

The other popular indentation method is the "indentation crack length" or "indentation fracture" method wherein a Vickers indentation is made on a polished specimen surface and the lengths of the corner cracks are used to evaluate fracture toughness.[15,16)] This method was never seriously considered for standardization. The method is popular in the ceramics community because of its apparent simplicity, the need for only one small piece, and the potential for making repeat measurements. In contrast, the traditional fracture mechanics community remained quite skeptical about the precracks and the need for a calibration constant (e.g., 0.016 ± 0.004).[b,16)] It is unreasonable to expect a wide range of ceramics to deform and fracture underneath an indentation in a similar manner. For example, a covalent bonded, hard ceramic (e.g., silicon carbide) deforms and fractures underneath an indentation very differently than an ionic bonded, cubic ceramic (e.g., magnesium oxide), or a glass. As the years have progressed, empirical fitting of different equations has continued unabated and a plethora of equations relating toughness to crack length have evolved adding to the confusion.[c,17)] The apparent simplicity of the method is compromised by experimental difficulties. A VAMAS round robin confirmed that estimates of the final crack tip position are highly subjective and equipment sensitive.[9,10)] Calculated fracture toughnesses varied by almost a factor of two for a fine-grained zirconiaalumina composite. So in summary, while the indentation fracture method may have some utility within a laboratory for research purposes, experience belies the method's suitability as standard method for producing accurate results.

The DCB method is held in high esteem by both the fracture mechanics and the ceramic communities, but it was not included since it differs considerably from the other three methods which utilize beams in bending. DCB is a leading candidate for future standardization.

The single-edged notched beam (SENB) method was never seriously considered, even with very thin (50~100 μm) diamond saw notches. This method has a propensity to overestimate fracture toughness and resvlts have a strong dependence on notch width. A blunt notch is not a sharp crack and thus the method does not meet one of the basic requirements of a fracture toughness test: the specimen should have a sound, well-defined crack. More recently, the single-edged V-notched beam (SEVNB) method wherein a SENB specimen is refined by polishing a very sharp notch into the notch root has shown some promise.[18,19)] Indeed, a full VAMAS round robin based on this method is currently underway. The early indications are that results from the SEVNB method may approximate true, sharp crack fracture toughness values to within 5% to 10%. Notch root bluntness and crack initiation phenomena may possibly account for the 5% to 10% difference.

The following sections of this paper cover the key elements of PS 070-97.

II. ASTM Standard PS 070: General

The core section of PS 070 includes general sections applicable to all three test methods. The details of the SEPB, SCF and CNB methods are included in three normative annexes. The draft document is over 70 pages in length,[d] which undoubtedly will seem unwieldy to some. The document is long since it includes three different methods, it includes many testing details and nuances that are intended *to help the users*, and it includes many extra elements (such as a precision and bias statement) not found in the JIS or DIN standards. JIS R 1607, for example, has nothing about possible R-curve behavior.

There are many notes and references to clarify requirements, to provide some rationale for a requirement, or to lead the user to additional published detail. The extensive detail is common ASTM practice. Detailed tolerances and procedral details are specified, not with the objective of "hamstringing" or forcing users to "do the test by our way", but to help users obtain successful, accurate, and precise results.

III. ASTM Standard PS 070-97: The Core Section

The core section of PS-070-97 includes many subtopics as discussed below.

1. Scope

The scope section states that the methods are primarily intended for use with advanced ceramics which are macroscopically homogeneous. Certain whisker-or particle-reinforced ceramics may also meet the macroscopic behavior assumptions.

2. Terminology

This section includes many critical definitions. Wherever possible, terms were taken from prior, conventional ASTM E-08 fracture standards. Otherwise, new definitions were created specifically for PS 070-97. Some example of the definitions are:

Fracture toughness: a generic term for measures of resistance of extension of a crack.

Slow crack growth (SCG): subcritical crack growth

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[c] Ponton and Rawlings[17] tabulated nineteen renditions in 1989 and there probably are many more by now.

[d] The final printed version may be ≈35 pages in length in the ASTM Annual Book of Standards.

(extension) which may result from, but is not restricted to, such mechanisms as environmentally-assisted corrosion or diffusive crack growth.

R-curve: a plot of crack-extension resistance as a function of stable crack extension.

Critical crack size: in this standard, the crack size at which the maximum load and catastrophic fracture occur in the precracked beam (see Figure) and the surface crack in flexure (see Figure) configurations. In the chevron notch specimen this is the crack size at which stress intensity factor coefficient, Y*, is at a minimum or equivalently, the crack size at which the maximum load would occur in a linear elastic, flat R-curve material.

Precrack: a crack that is intentionally induced into the test specimen prior to testing the specimen to fracture.

Small crack: a crack that is defined as being small when all physical dimensions (in particular, with length and depth of a surface crack) are small in comparison to a relevant microstructural scale, continuum mechanics scale, or physical size scale. The specific physical dimensions that define "small" vary with the particular material, geometric configuration, and loadings of interest.

Stable crack extension: controllable, time independent, noncritical crack propagation (Note – The mode of crack extension (stable or unstable) depends upon the compliance of the specimen and test fixture; the specimens and crack geometries; R-curve behavior of the material; and susceptibility of the material to slow crack growth).

Unstable crack extension: uncontrollable, time-independent, critical crack propagation.

It was ironic that this C-28 standard could not use the ASTM definition for K_{Ic} which has a very specific definition tailored to metal fracture toughness testing and standard E 399 in particular. This definition refers to specific operational requirements such as fatigue precracking and it is inappropriate for ceramics. To circumvent this dilemma, new fracture toughness terms were defined such as:

Fracture toughness K_{Isc}: the measured (K_{Isc}) or apparent (K_{Isc^*}) stress intensity factor corresponding to the extension resistance of a semielliptical crack, formed by Knoop indentation, for which the residual stress field due to indentation has been removed. The measurement is performed according to the operational procedure herein and satisfies all the validity requirements.

Comparable definitions exist for the SEPB and CNB methods.

3. Crack plane

The standard includes a scheme for reporting the crack plane and the crack extension direction, features not in the JIS and DIN standards.

4. Interferences: Slow crack growth, R-curve, and stability

The Interferences section discusses R-curve and slow crack growth phenomena, and also has a section on "stability". Stability is used in two contexts in PS 070-97. Stable crack extension is an essential prerequisite for a valid chevron notch test and the standard has specifications and requirements to ensure a proper test has been conducted. On the other hand, many fracture tests, such as the SCF method in the standard, are almost always unstable. Once the crack propagates, it does so in an uncontrollable manner.[e]

The alternate context for "stability" pertains to the possibility that stably-propagated cracks may have an intrinsically-different fracture toughness than that for unstably-propagated cracks, even for flat R-curve materials.[20)] The stiffness of the test system and the specimen may have an effect on the measured SEPB fracture toughness. SEPB tests are usually run unstably, but with careful fixture design and stiff testing machines, stable crack propagation is feasible. The ASTM committee decided not to delve too deeply into this topic but to at least bring it to the attention of the user.

5. Specimen requirements

Conventional 3 mm × 4 mm × ~45 mm flexure specimens are acceptable for all three methods, but the CNB and SEPB methods allow alternative configurations as well. Shorter SEPB specimens are also allowed. Generic specimen preparation requirements are in the core section, and specific additional details are in the three annexes. A minimum of four valid specimens are required, so more than four specimens should be prepared.

6. Flexure fixures

Generic requirements for the flexure test fixtures and other measurement equipment are included. The specimen type and fixture configurations are shown in Fig. 3. The SCF method is done in four-point flexure and 20 mm × 40 mm spans are recommended. The CNB test is conducted in either three- or four-point flexure, depending upon which one of the four specified specimen types is chosen. Outer spans again may be 38 mm to 40 mm. The SEPB test may be done in either three- or four-point flexure and a range of outer spans (typically 16 mm to 40 mm) is permitted.

Common 20 mm × 40 mm four-point flexure strength fixtures that are designed to be in accordance with the well-known standard flexure strength test standards[21)] are satisfactory for all three fracture toughness methods. Users are reminded that there *are critical requirements* for load pin and specimen alignment, articulation, and load pin rolling action to eliminate friction constraints in flexure testing.

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[e] The exception might be for instances of very steep R-curves at small creak sizes, but this is expected to occur over only short crack extensions and very quickly so that it world be difficult to control or monitor with conventional testing equipment during an SCF test.

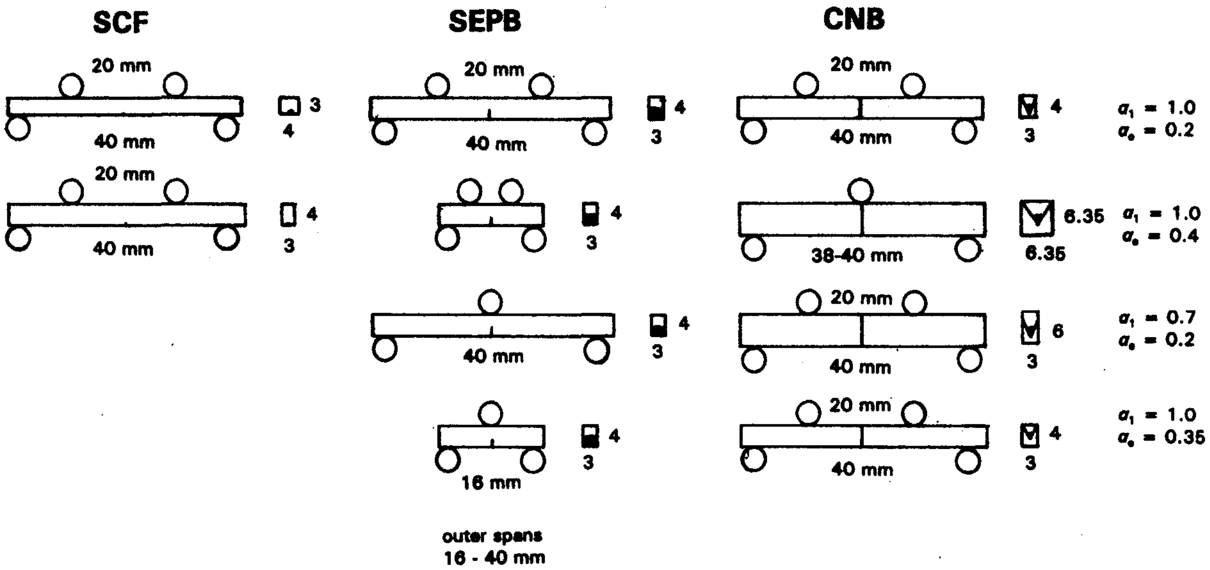

Fig. 3. The specimen and fixture combinations recommended or required in PS 070-07.

7. *Test rates and slow crack growth*

Testing rates between 0.1 MPa$\sqrt{m}$/s and 2.75 MPa$\sqrt{m}$/s are specified for normal testing. If SCG is suspected, the standard recommends that tests with two different rates (at least 2~3 orders of magnitude different) should be used. Alternately, testing in laboratory ambient conditions should be matched by parallel trials in an inert (e.g., dry nitrogen) environment.

8. *R-curve behavior*

If R-curve behavior is suspected, then the standard recommends that two of the three methods be performed and the results compared.

9. *Valid testing*

Reporting requirements round out the core protion of PS 070-97. The standard also requires a positive statement affirming that *all validity requirements* for the respective tests have been satisfied.

10. *Precision and bias*

A unique element in ASTM test method standards is a Precision and Bias statement. It is intended to give some guidance as to what to expect in terms of the accuracy and precision of test results done in accordance with the standard. Precision is the closeness of agreement between test results obtained under prescribed conditions. A statement on precision allows users of the test method to assess its usefulness in proposed applications. Within-laboratory precision is known as "repeatability" and between-laboratory precision is termed "reproducibility". Bias is a systematic error that contributes to the difference between a mean of a large number of test results and an accepted reference value.

Table 1. Precision Values for the SCF and SEPB Methods from two VAMAS Round Robins

Test Method	Material[f]	Repeatability* (Within-Lab Precision)	Reproducibility* (Between-Lab Precision)
SEPB[9,10]	gas pressure sintered Si_3N_4	4.6%	7.1% or 8.8%(**)
SCF[22,23]	hot-pressed silicon nitride grade NC 132[g]	5.4%	6.8%
SCF[22,23]	hot-isopressed silicon nitride grade Ekasin[h]	7.7%	8.9%
SCF[22,23]	yttria-stabilized ZrO_2	6.6%	6.6%

*Repeatability and Reproducibility standard deviations are expressed as coefficients of variation.
**Reproducibilities depended upon the crosshead rates.

Statistically derived precision estimates are given in Table 1 for the SEPB and SCF methods for which data from two VAMAS round robin projects were available.[9,10,22,23] The uncertainties were calculated in accordance with ASTM E 691[24] which is very similar to ISO 5725.[25] These standards give "repeatability standard deviation" and the "reproducibility standard deviation" which are shown in Table 1, except that the standard deviations have been converted to coefficients of variation.

A basic premise of PS 070-97 is that the three methods should produce the same results for materials with flat R-curves if there is negligible SCG. Example data from the three methods for sintered silicon carbide and

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[f] Certain commercial materials are identified in this paper to specify adequately the experimental procedure.
[g] Norton Co., Worcester, MA.
[h] ESK, Kempten, Germany.

hot-pressed silicon nitride are included in the standard's Precision and Bias section. The agreement is outstanding for the silicon nitride. Mean fracture toughness values for sample sizes of ~5 specimens concur within 0.1 MPa $\sqrt{m}$ to 0.3 MPa$\sqrt{m}$. Much of the variability is due to batch to batch variations in the ceramic. Recent work, described below in the section on the prototype SRM 2100 work, has consistently demonstrated that agreement of 1% or better (0.05 MPa$\sqrt{m}$) may be achieved between the three methods with sample sizes of 5 or more.

We conclude that the three methods indeed have been refined to the degree that they are capable of producing accurate and precise data. The three test method annexes are discussed below.

IV. Single-edged Precracked Beam Annex

The 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 SENB method, the SEPB method uses an innovative precracking procedure (compression bridge-anvil loading) to produce sharp precracks.[26] A saw cut or indentation is placed in the narrow side of a flexure specimen. The specimens then is placed into a compression-anvil precracking apparatus and loaded carefully until a precrack pops in. The specimen then in fractured in a flexure fixture. Crack size is measured on the fracture surface.

The SEPB annex has 12 pages of detail to help the user obtain valid outcomes. Either three- or four-point loading is allowed as shown in Fig. 3. A wide range of specimen and fixture sizes is allowed. Guidance on the bridge precracker design in furnished. Precracking loads depend upon a variety of factors such as the fixture and specimen geometries and their material properties.[27,28] In practice, well-machined, flat and parallel bridge precrackers are essential. The pop-in load and the resultant crack depth can be adjusted by the choice of different sized precracker grooves.

Stress intensity factor solutions are provided for fixture outer span lengths to specimen height (S_o/W) ratios from 4 to 10. Thus, for a conventional 4 mm wide specimen, fixture spans from 16 mm to 40 mm are allowed. Traditional stress intensity factor coefficients are used for three-point and four-point configurations, but new refined coefficients are used for S_o/W ratios of 5 or larger.[20] Very rigid test set ups (testing machine and fixtures) may be advantageous in some instances if crack extension stability is a factor in the outcomes. There is a strict requirement that the indentation load *shall not exceed 98 N* since there is ample evidence from several sources (e.g. see Refs. 27 and 29) that larger indentation loads will create residual stresses that affect the measured fracture toughness. The size and straightness of the precrack front must be verified. The initial crack depth (a) to specimen width (W) ratio, α=a/W, must be between 0.40 and 0.60. This range will be extended to 0.35 to 0.60 in the final consensus version of the standard. There is reuctance to extend the lower bound to 0.30 (the limit in JIS R 1607) since there is ample experimental evidence in the literature[27,29-33] that residual stress interferences from the indentations may affect the results adversely at short ($\alpha \approx 0.3$) crack lengths.

There are a few other differences between ASTM PS 070-97 and JIS R 1607. Our own experiences indicate as much as a 10% difference of the minimum crack depth compared to the mean crack depth is tolerable.[27] This tolerance is more relaxed than that in JIS R 1607, which requires the maximum versus minimum crack depths differ by no more than 10%. Crack twists or misalignments of up to 5° are permitted in the ASTM standard for 3- or 4-point loading.[i] Twists of up to 10° are allowed in JIS R 1607.

During the SEPB fracture test, load versus specimen displacement, or actuator displacement, or backface strain,[34,35] or time must be recorded. Back-face strain monitoring, in which a strain gage is applied to the specimen face opposite the crack, is a new means to detect stable crack extension. Caution should be exercised to recognize whether the precrack may have extended during the fracture test. At the present time, the standard uses the initial precrack size in conjuction with the maximum fracture load. If there is stable crack extension, then the true crack size at instability is longer. Evidence for stable crack extension may be from the data taken during fracture testing or from fracture surface markings.

The VAMAS SEPB round robin[9,10] produced valuable information for this method including the precision estimates mentioned earlier. One of the common complaints about this method is that it is difficult to precrack the specimen. Some of the participants in this and other VAMAS round robins reported very low precracking success rates. The problem seemed to be in the design and tolerances of the precracking fixtures. Well-designed, optimized precracking fixtures usually have 80~100% success rates. Alignment in three-point loading of short specimens in flexure fixtures also proved to be problematic. The most recently completed VAMAS round robin[36] that used this method detected a perceptible shift (0.7 MPa$\sqrt{m}$, or 10% overestimate) in toughness values for 3-point, 16 mm span outcomes relative to larger span 3-point or 4-point results. In our preparation work for SRM 2100 at NIST (described below), there were problems aligning precracks within the required 0.5 mm of center. Misaligned cracks were more apt to twist or tilt, which led to overestimates of fracture toughness.

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[i] This specification will be changed to 10° for 4-point loading in the full-consensus ASTM standard, but will remain at 5° for 3-point loading.

V. Surface Crack in Flexure Annex

The SCF method, also known as the "controlled flaw" method, is a development of early work by Petrovic and colleagues.[37] This innovative method utilizes a Knoop indenter to create a tiny, semi-elliptical surface precrack.[j] Residual stresses from the indentation must be eliminated by polishing or hand grinding the indentation away after indentation. Fractography is used to measure the precrack after fracture. The SCF method will not work on soft or very tough ceramics, since precracks do not form under the indentation. The method also will not work in some ceramics in which fractography is difficult. The easiest way to check this is to precrack one specimen then break it, without removing the indentation and residual stress zone by polishing. The fracture surface may be inspected and it may be confirmed that fracture has occurred from the precrack and whether the precrack is detectable.

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 the name consistent with fracture mechanics conventions. For example, ASTM Committee E-08 has an analogous standard for metals, E 740[38] called the "Surface Crack in Tension (SCT)" method. In the SCT test, a semielliptical surface carck is machined into a common tension strength specimen. Fatigue loading is used to extend and sharpen the crack. Newman-Raju stress intensity factor coefficients (Y)[14] are used to compute the fracture toughness in the SCT method.

The surface crack in flexure (SCF) test method in C-28's standard PS 070-97 is conceptually the same, but instead of machining a precrack, a Knoop indenter makes a tiny semi-elliptical surface flaw. The flaw is a little larger than the natural flaws and cracks in the ceramic. The essential step of removing the residual stresses is best done by hand grinding. Annealing is not recommended since there are risks of crack healing or crack tip blunting. After precracking and removal of the residual stresses, the specimen is fractured in flexure and the precrack size and shape measured on the fracture surfaces. The same Newman-Raju Y factors that are in ASTM E 740 are also used in PS 070-97. Fracture toughness is computed:

$$K_{Isc} = Y\sigma\sqrt{a} \qquad (1)$$

where Y the stress intensity shape factor, σ is the maximum stress in the flexure specimen, and a is the crack size. Y is a simple function of crack depth, a, and width, 2c.[k]

Extensive work to refine the SCF method[22,23,39] and successful outcomes in the VAMAS round robin[22,23] provided the impetus for standardization of this method in PS 070-97. PS 070-97 includes considerable detail on precrack implantation, residual stress removal, specimen fracture, and fractographic analysis and interpretation. The detail is the result of years of refinements to this method and, again, is intended to aid but not hamper the user with excessive detail. The SCF annex of PS 070-97 runs twelve pages in length.

The hand grinding step is a drawback to this method, but information is furnished on how to accomplish this efficiently. The requirement for fractographic analysis is also a drawback. Two supplemental appendices with eight pages of illustrations and photos are included to aid interpretation. Precrack detection is difficult in some materials, and decoration methods are under development. Nonetheless, the VAMAS round robin showed that some members who had never tried the method before and who had limited prior fractographic experience obtained good results.

Indeed, one of the extraordinary findings of the VAMAS project was that the method is *not highly sensitive* to the crack size measurements. The source of this "forgiveness" is due to the interaction between "a" and "Y" in equation 1. An erroneous estimate of crack size is mitigated not only by the square root dependence of toughness upon crack size (equation 1), but also by a *compensating adjustment in the calculated Y factor*. That is to say, an overestimate in crack size, a, is matched by an underestimate in the computed Y. For some crack configurations, a 10% error in the crack size measurements leads to as little as a 1% error in fracture toughness. The key is that an individual *Y must be computed for each and every precrack*. An average or "effective value" is not sufficient.

The SCF test method procedure provides guidance for instances wherein slow crack growth or stable crack extension does occur. If it is discerned on the fracture surface, the standard recommends that fracture toughness be computed on the basis of both the initial crack size, K_{Isc}, *and* the crack size at instability, K_{Isc}*. If there is any doubt about the interpretation, then inert atmosphere testing or fast testing rate experiments should be done. Recent findings confirm that SCG markings can be detected on SCF precracks in some materials and fracture toughness may be calculated based on the crack size at instability.[40,41]

SCF results are representative of the toughness that natural flaws experience when loaded to fracture, in contrast to the large-crack methods. For brittle ceramics that have negligible R-curve behavior, it is expected that

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[j] Of the three test methods in PS 070-97, the SCF method is the least familiar to the general fracture mechanics community. Although microindentation methods are viewed with some skepticism by the latter, the similarity of the SCF and SCT methods alleviated these concerns.

[k] Y will be a maximum at either the deepest part of the precrack periphery, or where the crack intersecets the tensile surface. Both Y values are calculated and the maximum is used in equation 1.

the SCF method will give results identical to the SEPB and CNB methods. Data are furnished in the precision and bias section of PS 070-97 that bears this out. In instances for materials with rising R-curve behavior, it is expected that the SCF fracture toughness will come from the small crack portion of the R-curve. The fracture toughness may better represent the local fracture toughness that a natural flaw in the ceramic will experience. It may be possible that an "effective R-curve" could be generated for a ceramic by varying the initial indentation load, which will produce precracks of varying sizes. This is a topic worthy of future investigation.

VI. Chevron Notch in Bending Annex

The CNB annex is nine pages long. This method entails loading a chevron-notched specimen to fracture in a controlled fashion in a flexure fixture. The crack initiates at the chevron tip and then must propagate stably through the chevron. Four *very specific* CNB specimens shown in Fig. 3 are prescribed in the standard since generalized stress intensity factor solutions for a variety of chevron notch specimens do not currently exist. Three- or four-point loading is used depending upon which chevron notched specimen is chosen. Stringent requirements are placed upon the notch preparation. Misalignments in the chevron will promote unstable crack propagation that will invalidate the test.[42] Stress intensity factors are very sensitive to the precise geometries as well. Analytical errors of 10% or more are possible if formulas for idealized chevron geometries are applied to poorly-fabricated specimens. Consequently, the tolerances on the chevron notch have been chosen with the objective of not only inducing stable crack propagation, bot also for maintaining the geometry within reasonable acceptable bounds (low error) for the Y* analytical expressions. It is especially important that:

a. the two notch grooves (one on either side of the specimen) meet very closely without an offset,

b. the notch tip be well centered,

c. the sides of the chevron be very symmetric about the specimen centerline,

d. the sides of the chevron 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. 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 next cut made from the opposite side while the specimen is held in a precise location.

The four geometries in the standard were chosen based on prior experience and good stress intensity factor solutions were available based upon Bluhm's slice model.[43] Since generalized relations for chevron notch stress intensity factor coefficients (Y*) do not exist, specific expressions are included for the four geometries. The Y*'s are sensitive to the precise geometric details of the chevron. *Any deviations from the assumed configuration introduce analytical errors.*

Recent work[44] at NASA-Lewis has shown that a straight through crack assumption (STCA) model gives better agreement with finite element results for chevron configurations A, B, and D in PS 070-97. Consequently, the full consensus version of the ASTM standard will substitute new Y* solutions for these three configurations. The changes in calculated fracture toughness are typically a few percent. This is the most important technical revision to PS 070-97.

Crack propagation for a CNB test must be stable for the result to be valid. Consequently, the standard requires monitoring load versus any one of specimen edisplacement, load-point or actuator displacement, back-face strain,[34,35] or time. The standard includes illustrations of unstable (invalid) and fully-stable (valid) load-displacement curves, as well as the commonly observed pop-in from slight overload followed by stable extension (valid) behavior that is commonly obtained.

PS 070-97 also requires that the fracture surfaces be examined to ensure that the crack properly followed the notch plane. As with the other two methods, if SCG is suspected, then additional experiments in inert atmosphere or at different rates are recommended.

VII. Full-consensus Standard in 1999

A full-consensus version of the standard is still being developed, but the sense is that the document is in a final polishing stage. There are over 100 changes in the full-consensus version compared to PS 070-97! A list of the revisions to help users keep track of the differences has been maintained. Most changes are very small editorial corrections, but the following revisions are substantive:

a. The Y factors for the 3-point SEPB configuration with a span to width (S_o/W) ratio of 5 were refined.

b. The tolerance on centering a SEPB precrack in 4-point fixtures was relaxed from ±0.5 mm to ±1.0 mm.

c. The tolerance on the crack twist in SEPB has been relaxed from 5° to 10° for 4-point loading, but remains 5° for 3-point loading.

d. The allowable SEPB precrack depth range has been broadened from (0.40 to 0.60) to (0.35 to 0.60).

e. The CNB Y* factor equations were changed. STCA factors are used for configurations A, B, and D.

f. Backface strain was defined.

g. The amount of material to be hand ground from SCF specimens was increased slightly from "4.3h to 4.5h" to "4.5h to 5.0h" where h is the depth of the indentation.

h. Paragraphs on validity requirements were added to each test method section.

i. More computation examples were added.

For the moment the full consensus standard will still include the very short span (16 mm) 3-point SEPB configuration although we have growing concerns about the difficulty in conducting these tests and obtaining proper results. It is included since it is a traditional fracture mechanics configuration from the metals community. (A scaled-up version is standardized in E 399.) The configuration also is in JIS R 1607. As noted above, there is a growing body of literature (e.g. Refs. 27 and 45) that indicates that faulty results may be obtained with the very short span 3-point specimens. It certainly is a nuisance to meticulously slign the precrack (or the precrack tip?) with the middle loading roller. On a practical basis, 4-point specimens on 10 mm×20 mm or 10 mm×30 mm spans are much simpler to test, since there less care is required for alignment, and the test results are probably more accurate.

VIII. Standard Reference Material 2100

NIST is preparing Standard Reference Material (SRM) 2100 which will have certified values of fracture toughness. This SRM will be available in 1999 in the form of packets of 5 NC 132 silicon nitride specimens for a nominal price ~$335. Each packet will include a NIST certificate with the certified average value of toughness that should be obtained with the five specimens. The specimens will be 3 mm×4 mm×45 mm conventional flexure specimens that will have to be precracked by the users. The choice of precracking procedure and test method will be left to the user. Specimens may be fractured multiply. That is, the first test may be SCF, SEPB, or CNB, and then the halves may be fractured again. One face of the specimen has been fine ground by a 900 grit wheel for the convenience of users who may wish to use the SCF method. The fine ground surface is suitable for Knoop precracking. Over 1000 specimens have been prepared from 6 billets of hot-pressed silicon nitride, grade NC 132. Over 200 specimens were tested using the procedures of PS 070-97 as well as some variants. These experiments comprise the data base that will be used to certify the remaining untested specimens.

This SRM had its roots in the VAMAS fracture toughness by the SCF method round robin.[22,23)] There was a remarkable consistency in results for the hot-pressed silicon nitride and these results concurred with data that had been generated by many laboratories over 20 years. In particular, results by the three test methods in PS 070-97 converged in the range of 4.5 MPa$\sqrt{m}$ to 4.9 MPa$\sqrt{m}$.

To further explore this, new fracture toughness test specimens subsequently were prepared from six separate billets of NC 132, and these were tested in accordance with the three methods in PS 070-97. Table 1 shows the results, which are in extraordinary agreement: for any particular billet the mean fracture toughness for sets of 5 specimens usually agrees within $\approx 1\%$, *irrespective of test method*. The fracture toughness varied very little within a billet, but there were distinct differences between billets. Even the standard deviations were consistent.

This 1970~1980's vintage material, which was hot-pressed with a small amount of magnesium oxide sintering aid, is composed of nearly 100% beta silicon nitride grains with only a slight elongation. The small amount of amorphous second phase resides in small triple point pockets or as thin layers between the silicon nitride grains. Some tungsten impurity compounds are distributed throughout the bulk. NC 132 fractures in a mixed transgranular and intergranular mode at room temperature. It has a flat R-curve and is highly resistant to environmentally-

Table 2. Proliminury Fracture Toughness Data for 6 Hot-pressed Silicon Nitride Billets by the three Test Methods in PS 070-97. There is an Extraordinary Consistency of the Fracture Toughnesses by the three Methods within any Billet

Billet	Method	Mean*	Std. Dev.*	Comments**
C	SCF	4.58	0.16	26 valid outcomes from 6 sample sets
C	CNB	4.60	0.13	8 valid outcomes, 3- or 4-point loading; chevron configuration "A"; STCA Y* polynomials; tested at NASA-Lewis
C	SEPB	4.58	0.10	19 valid outcomes from 4 sample sets
D	SCF	4.53	0.22	19 valid outcomes from 5 sample sets
D	SEPB	-	-	(data unavailable)
G	SCF	4.29	0.19	15 valid outcomes from 3 sample sets
G	SEPB	4.27	0.16	15 valid outcomes from 3 sample sets
4	SCF	4.34	0.19	10 valid outcomes from 2 sample sets
4	SEPB	4.37	0.07	4 valid outcomes from 1 sample set
A	SCF	4.28	0.21	28 valid outcomes from 6 sample sets
A	SEPB	4.14	0.30	26 valid outcomes from 6 sample sets
H	SCF	3.87	0.09	5 valid outcomes from 1 sample set
H	SEPB	3.85	0.08	valid outcomes from 1 sample set

*MPa $gfonrr\sqrt{m}$. **A sample set comprised either 5 or 6 specimens.

assisted slow crack growth at room temperature. Consequently, it should not be surprising that the three test methods in PS 070-97 produce virtually identical toughness results.

Billets A and H appear to be atypical. The scatter in fracture toughness in billet A is too large and some faint color variations were detected in the specimens. This billet will not be utilized for the SRM. Billet H specimens showed clear evidence of SCG on the SCF specimen fracture surfaces in specimens tested under lab ambient conditions (Table 2 data). Five specimens were tested in dry nitrogen (just as PS 070-97 recommends) and the evidence of SCG disappeared. The dry nitrogen SCF average toughness was 4.57 $\mathrm{MPa\sqrt{m}} \pm 0.17\ \mathrm{MPa\sqrt{m}}$. Thus, it appears that billet H is susceptible to SCG under laboratory ambient conditions.

This data consistency confirms that the PS 070-97 methods for measuring fracture toughness have been optimized to the extent that genuine material variability (billet-to-billet, or batch-to-batch) can now be discerned. This is one goal of any standard: to create and refine tools that can be used to measure the desired property with good accuracy and precision.

The 200+ specimens tested specifically for the SRM data base, plus 85 earlier experiments at NIST, and the $\approx$220 specimens tested during the VAMAS round robin, constitute over 500 fracture toughness experiments on this particular material.

IX. Conclusions

Three mature test methods, CNB, SCF and SEPB, have been incorporated into the new ASTM standard test method PS 070-97, which soon will be converted into a full-consensus standard. These three methods produce accurate and precise results when the conditions for validity have been met. VAMAS round robin data has verified these claims and furnished very specific estimates of the precision of two of the three methods. A full-consensus standard that is nearly identical to PS 070-97 is currently balloting in Committee C-28, Advanced Ceramics, and will likely be adopted in early 1999. NIST SRM 2100 is being prepared to support the test method standard. The SRM will enable users to apply the test methods with confidence and will enable laboratories to establish their own accuracy and precision estimates. The extraordinary consistency of results validates the three test methods and demonstrates accuracy and precision of the order of 1% or better (coefficient of variation) may be achieved by add certifying laboratory for a monolithic ceramic. The VAMAS round robins showed that between laboratory reproducibilities of 5% to 9% (coefficient of variation) are achievable.

As is usual with any method, a little experience enhances the chances of success with the methods. We anticipate that as experience is gained, the standard will be revised, corrected and expanded particularly to deal with R-curve phenomena. New test methods may be added as annexes or as new standards altogether.

We end by noting that there always will be new methods and procedures under development which offer the prospect of simpler, faster, more efficient testing. We welcome these developments but prudence suggests that comprehensive testing to establish the robustness of the new methods nd to evaluate their general applicability, accuracy, and reliability is a prerequisite for standardization. In fracture mechanics testing, there are few shortcuts. There are no substitutes for methods based on sound fracture mechanics and metrological principles.

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