FRACTURE TOUGHNESS OF CERAMICS BY THE VICKERS INDENTATION CRACK LENGTH METHOD: A CRITICAL REVIEW

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ABSTRACT

Fracture toughness is an important property that characterizes a material's brittleness or resistance to fracture. Although some fracture toughness test methods have been refined and even standardized, many researchers have continued to use the Vickers indentation crack length method as an expedient. In this paper, Vickers indentation fracture resistance data for Standard Reference Material SRM 2100 are presented and the weak points of the method are reviewed.

INTRODUCTION

The state of the art of fracture toughness testing has significantly improved in recent years. Several methods have been painstakingly refined and optimized. ASTM International,[1] Japanese Industrial Standards (JIS),[2] and European Committee for Standards (CEN)[3] formal standard test methods are available for the single-edged precracked beam (SEPB), chevron notched beam (CNB), and surface crack in flexure (SCF) methods. These methods have single well-shaped precrack, a good loading configuration, and an accurate stress intensity factor solution. They are now approved as standards by the International Organization for Standards.[4,5] International round robins have confirmed that accurate and precise data are obtained with these methods.[6] Other test methods such as double torsion and single-edged V-notched beams (SEVNB) currently are being developed as standards. Those who have developed and refined these test methods have striven to make them technically rigorous, accurate, precise, and practical.

In the 1970s and 1980s there was considerable confusion about fracture toughness testing. Results were quite variable and seemed to depend upon the test method. Slow crack growth and R-curve phenomena contributed to the data variability. Some even wondered whether there was such a thing as a set value for fracture toughness, K_{Ic}, since all tests seemed to give different outcomes. For example, it was common to hear that "large crack" fracture toughness specimens gave different outcomes than "short crack" specimens. The test methods were gradually refined and the signs of slow crack growth and /or R-curve behavior were recognized. Testing procedures were adapted to either minimize their effects or to study them. Despite these complications, it is now known that many brittle materials do have a specific fracture toughness in the classical sense. These include fine-grained polycrystalline ceramics and coarse-grained ceramics that fracture transgranularly without bridging. For example, pressureless sintered alpha silicon carbide with boron and carbon sintering aides has no boundary phase. It fractures transgranularly and has a fracture toughness of about 2.5 to 2.9 MPa$\sqrt{m}$ depending upon the density and batch and the result does not depend upon the test method.[1]

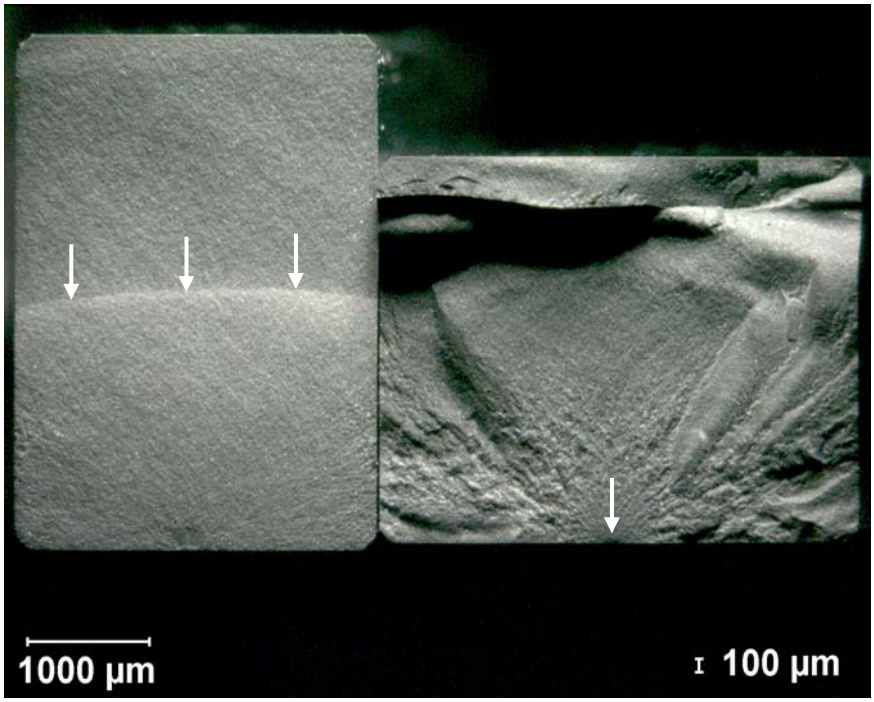

Figure 1 Fracture surfaces of two SRM 2100 silicon nitride bend bar specimens. The SEPB test piece on the left had a ≈ 2 mm deep precrack (arrows), whereas the SCF test piece on the right had a ≈ 50 µm deep semielliptical precrack (arrow). Identical fracture toughness outcomes are obtained with these methods despite the dramatic differences in precrack size and shape.

Standard Reference Material SRM 2100[7] was prepared by NIST in the late 1990s.[8,9] It is the first standard reference material in the world for the property fracture toughness. SRM 2100 is a set of silicon nitride bend bars that are certified to have a specific K_{Ic} irrespective of test method. All told, five hundred and eighty experiments were done to prepare it including hundreds of experiments in an international round robin. Figure 1 shows the fracture surfaces of two specimens, with dramatically different precrack types and sizes, but which produced virtually identical fracture toughness outcomes.

The "Vickers indentation fracture, (IF)" or "Vickers indentation crack length" method is a nontraditional method. It does not break a precracked specimen. Instead, it uses a Vickers indenter to make a hardness impression on a polished specimen surface. The indenter creates a plastically-deformed region underneath the indenter as well as cracks that emanate radially outward and downward from the indentation. The cracks are assumed to be semicircular median cracks in most of the analyses. On the polished surface one sees four cracks that radiate outward from the indentation corners. The lengths of these cracks are measured. Fracture toughness is computed on the basis of the crack lengths, the indentation load, the hardness, the elastic modulus, the indentation diagonal size, and an empirical fitting constant. This method has the appeal that it uses conventional hardness equipment, uses a very small test piece, and does not require elaborate precracking or testing equipment. The method has a very poor track record, however. Much of the fracture toughness data produced by this method has been unreliable, inaccurate, or imprecise. Nevertheless, expediency has led many to resort to its use. It even is

used in some new or draft material specifications.[10,11,12] The method was never intended to be used in this fashion. The traditional fracture mechanics community has been skeptical of the Vickers IF method. There are concerns about the complexities of the precracks, the forces acting upon them, and the need for empirical calibration constants. The purpose of this paper is to alert users to weak points of the method, particularly with regard to its accuracy.

BACKGROUND OF VICKERS INDENTATION FRACTURE (IF) METHOD

The method was an outgrowth of indentation fracture studies in the mid 1970s through the 1980s. Many of the studies were primarily conceptual and had limited practical value until 1976 when Evans and Charles[13] used a dimensional analysis and empirical curve fitting to correlate the crack length (c) and indentation size (a) to fracture toughness. Their short paper included a graph of normalized fracture toughness versus the crack length ratio (c/a). This graph could be used to estimate fracture toughness. It also showed that the c/a ratio should be 2.5 or greater. They work was corrected and refined by Marshall and Evans[14] in 1981 with the publication of a new simplified formula:

$$K_c = 0.036\ E^{.4}\ P^{.6}\ a^{-.7}\ (c/a)^{-1.5} \tag{1}$$

In this paper, the indentation fracture toughness estimates by the Vickers indentation technique will hereafter be termed K_c or K_{ifr}. Other work culminated in two back-to-back Journal of the American Ceramic Society papers by Anstis, Chantikul, Lawn, and Marshall in 1981.[15,16] The first had refinements to the Vickers crack length procedure and the second presented a variation whereby indentations were used to make strength controlling flaws in bend bars. Their Part 1 paper[15] is the most widely quoted source on the Vickers IF procedure although Niihara's papers are also widely mentioned.[17,18] The models assume that the median crack forms, propagates, and then decelerates to its final configuration during the indentation cycle. The plastically-deformed residual stress damage zone underneath the indentation is assumed to behave as an expanding cavity in a solid. The expanding cavity stress distribution is converted to an equivalent force that pulls the median cracks apart. The formula for fracture toughness in units of $N/m^{1.5}$ from Anstis et al.[16] is:

$$K_c = 0.016 \left(\frac{E}{H}\right)^{0.5} \left(\frac{P}{c^{1.5}}\right) \tag{2}$$

where E is the elastic modulus in GPa, H is the hardness in GPa, P is the indentation load in N, and c is the crack length in m. The first two terms are sometimes combined as:

$$\chi = 0.016 \left(\frac{E}{H}\right)^{0.5} \tag{3}$$

Geometrical effects and other terms are rolled up into the dimensionless calibration constant (0.016). Anstis et al.[15] mentioned the uncertainty of this constant was ± .004, a substantial variability (25 %), but they did not indicate what the uncertainty really meant. If it is one standard deviation, then deviations greater than .004 value might be expected to occur frequently. The residual stress factor χ is assumed to be constant for a material and characterizes the opening effect of the elastic/plastic stress field on the median crack. The $P/c^{1.5}$ ratio in equation 2 bears some similarity to a classical fracture mechanics solution for a penny shaped

crack of radius c with opposing forces F pulling directly on the crack faces. The stress intensity K_I for the latter case is proportional to $F/c^{1.5}$. Much of the original derivation of equation 2 was concerned with correlating the indentation force P to an effective F which acts to open the median cracks that are assumed to form underneath the indentation.

It is worth mentioning here that one must be careful about the systems of units and also the choice of hardness in the various Vickers IF equations. The usual convention in the hardness community, and the one that is adopted in every formal hardness standard in the world, is to define Vickers hardness (HV) as the load divided by the contact area of the four faces of the indentation. This leads to the standard definition:

$$HV = 1.8544 \frac{P}{d^2} = 0.4636 \frac{P}{a^2} \tag{4}$$

where P is the indentation load, and d = 2a is the indentation diagonal size. On the other hand, many indentation fracture advocates defined hardness as the load divided by the projected surface area of the indentation:

$$H = 2.0 \frac{P}{d^2} = \frac{P}{2a^2} = 0.5000 \frac{P}{a^2} \tag{5}$$

The latter gives a 7.9 % greater hardness number. Although some contend that the latter formula is a better estimate of the "average" contact pressure underneath a Vickers indentation, it should be obvious that the contact pressures under the sharp Vickers indenter are very nonuniform and an average pressure is at best an approximation.

Many of the indentation fracture equations use hardness in very general terms. It is meant to characterize the deformation resistance and plastic flow behavior under the indentation. H and HV both depend upon the indentation load in a trend called the indentation size effect (ISE).[19,20] Hardness varies appreciably with indentation load at low loads, but the load dependence diminishes or is eliminated at loads of 20 N or greater. Most indentation fracture analyses have glossed over this point and the usual convention is to simply use a high load, plateau value for hardness in the equations. It is pointed out that the H dependence is small due to the 0.5 or 0.4 power it is raised to in the equations. Nevertheless, many of the IF equations are rewritten in an expanded form with the indentation load (P) and diagonal half length (a) replacing the hardness term. Thus, if equation 4 is substituted into equation 2:

$$K_{ifr} = 0.0226 E^{.5} P^{.5} a^{1.0} / c^{1.5} \tag{6}$$

Niihara developed a slightly different equation[17,18] for median cracks:

$$K_{ifr} = 0.0334 \left(\frac{E}{H} \right)^{0.4} \left(\frac{P}{c^{1.5}} \right) \tag{7}$$

and if equation 5 for hardness H is substituted:

$$K_{ifr} = 0.044 \; E^{.4} \; P^{.6} \; a^{.8} / c^{1.5} \tag{8}$$

A variant of Niihara's equation 7 is one option in the ASTM bearing ball specification F 2094:

$$K_{ifr} = 10.4\ E^{.4}\ P^{.6}\ a^{.8}\ /\ c^{1.5} \tag{9}$$

This expression requires that P be in kgf, a and c in micrometers, and E in GPa. The conversion factor in equation 9 is based on equation 4 for the HV hardness conversion. In equations 6, 8, and 9, users are able to insert indentation loads and sizes from small indentations without being aware that they are introducing variations due to the ISE.

JIS R 1607 for fracture toughness features the respected single-edged precrack beam method for determining K_{Ic}, but it also includes a Vickers IF method as an alternative to compute a "K_c" parameter. The formula, which is known as Miyoshi's equation,[21] is the same as equation 2 except that the constant is 0.018 and the hardness in the equation is HV and not H:

$$K_c = K_{ifr} = 0.018 \left(\frac{E}{HV} \right)^{0.5} \left(\frac{P}{c^{1.5}} \right) \tag{10}$$

This formula gives K_{ifr} values 17 % larger than equation 2. Users should also be cautious about the units in the equations. Some use Newtons for force, but others use kilograms force (kgf). Elastic moduli may be either in GPa, Pa, or N/m^2. Hardness may be in GPa or kgf/mm^2. Lengths may be in micrometers or meters. Conversion factors such as 9.8, 1000, and 10^9 may be raised to powers such as 0.4, 0.5, or 0.8 and combined into the calibration constant.

Even at large loads, for identical input equations 2, 6, 7, 8, and 9 produce K_{ifr} values that differ by as much as 50 %! There are many more IF equations, but for the moment we concern ourselves with the above variants since they are creeping into standards and specifications. Do they give accurate and reliable results or do they produce simple ranking estimates? To answer these questions, Vickers IF experiments were done on a NIST SRM 2100 test piece.

EXPERIMENTAL PROCEDURE

SRM 2100 is certified for a fracture toughness of 4.57 MPa√m ± 0.23 MPa√m. The uncertainty is the 95 % confidence interval for a single test outcome. The uncertainty shrinks to only 0.11 MPa√m for the average of five readings. The test piece was one half of a bend bar that had been used for a surface crack in flexure experiment. The 3 mm x 4 mm x 25 mm fragment was mounted in epoxy and polished to an optical finish. A 4 mm x 25 mm surface was used for the indentations. In hindsight, it may have been better to mount the specimen so that a 3 mm x 25 mm face was polished. The SRM is a hot-pressed silicon nitride that had a slight preferred orientation. This was duly noted in the SRM certificate.

All indentations were made with a Zwick model 3212[a] hardness tester but were measured with an optical microscope described below. The indentation contact time was 15 s. A NIST Standard Reference Material 2831 tungsten carbide hardness reference disk was used to verify the machine was operating correctly.[22] The average diagonal size for five indentations made at 9.8 N was 34.5 μm ± 0.3 μm, in excellent agreement with the certified value of 34.6 μm for the disk. Ten indentations were made in the silicon nitride SRM 2100 piece at loads from 9.8 N to 98 N. The latter load was large enough to generate the desired crack pattern. Greater loads such as 196 N or even 294 N are sometimes recommended, but these loads will cause severe chipping and shattering for many ceramics such as silicon carbide and boron carbide. Two preliminary indentations were also made at 4.9 N. Vickers hardness of the SRM material decreases with

[a] Certain commercial materials or equipment are identified in this paper to specify adequately the experimental procedure. Such identification does not imply endorsement by the NIST nor does it imply that these materials or equipment are necessarily the best for the purpose.

(a)

(b)

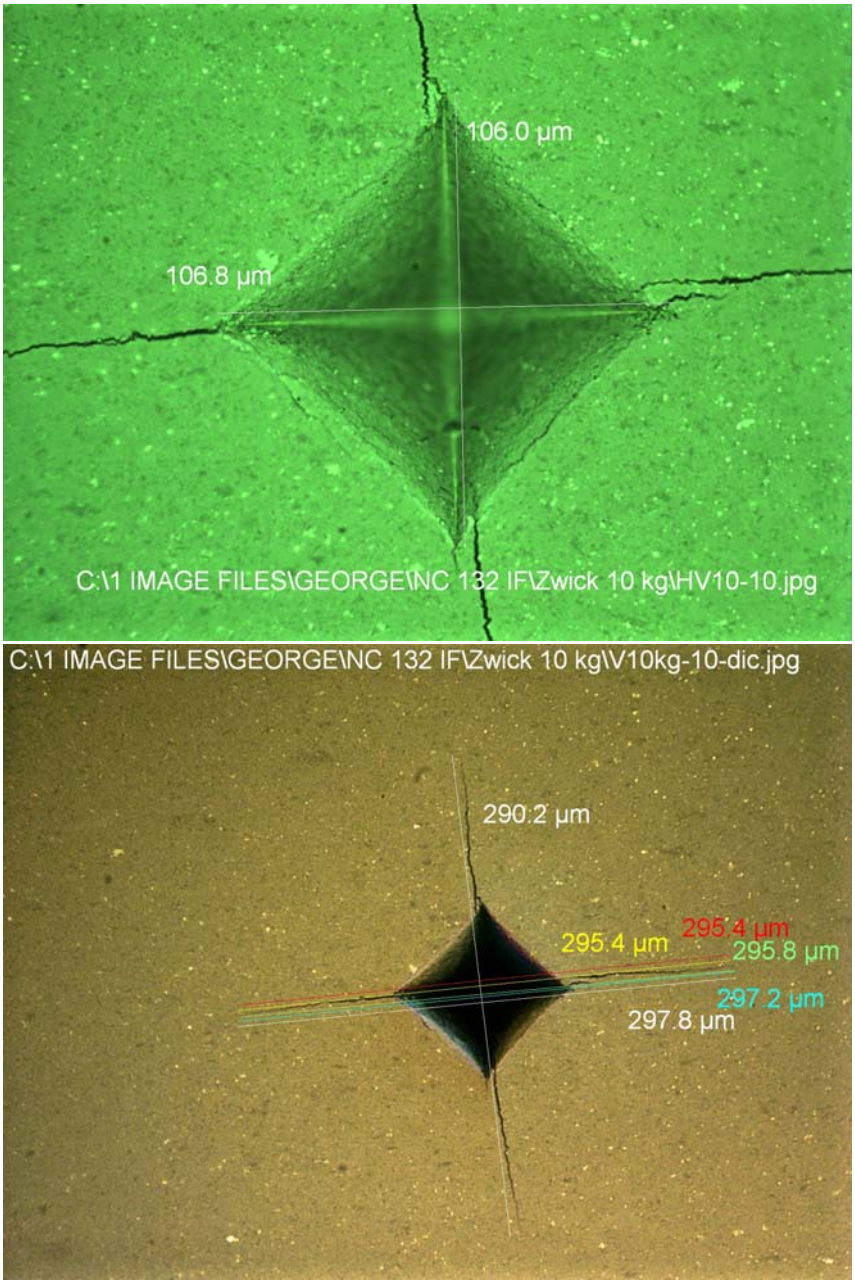

Figure 2 Two images of the same 98 N indentation in SRM 2100 silicon nitride. (a) shows a bright field image with a green filter and 40 X objective with auxiliary 1.6 X internal magnifier. The diagonal lengths (2a) differed in this instance by only 0.8 µm. Notice the slightly ragged edges and interior surface roughness due to individual grains protruding into and being shifted along the indentation contact area. These also affect the tip shape. Notice also the convenient tiny reflective spots from inclusions and some grains. These served as helpful markers when judging crack maximum lengths. (b) shows an interference contrast image with a 20 X objective. The horizontal crack length (2c) was measured five times for a repeatability standard deviation of 1.1 µm for one measurement. The multicolored measurement lines are only 1 pixel wide and may not reproduce well here.

increasing load, but reaches a plateau between 19.6 N (2 kgf) and 49 N (5 kgf).

All indentation sizes and the crack lengths were imaged and measured with a Leica DMRM research compound optical microscope.[b] Examples of the images are in Figure 2. This optical microscope is typical of conventional reflected microscopes that are commonly found in laboratories. A Diagnostics Instruments Spot Insight digital camera was mounted to the microscope and used to capture the images. The camera software displayed the indentations on a high resolution computer monitor. Sizes were measured on the computer monitor with a readout resolution of 0.1 μm. A very accurate and precise stage micrometer was used to verify the magnifications and size calibration factors under all illumination and magnification conditions. A variety of illumination and viewing schemes were evaluated in order to determine which were best for viewing the indentation size and the crack lengths. The best viewing modes for the indentations and the cracks were different. Figure 3 shows some indentation details.

The best clarity for measuring the crack lengths (2c) was with differential interference contrast illumination using a 20 X, 40 X, or 100 X objective as required to fit the image on the screen (Fig. 2b). The image of the indentation was projected onto the computer monitor, but the actual determination of a crack tip location was made while viewing through the microscope

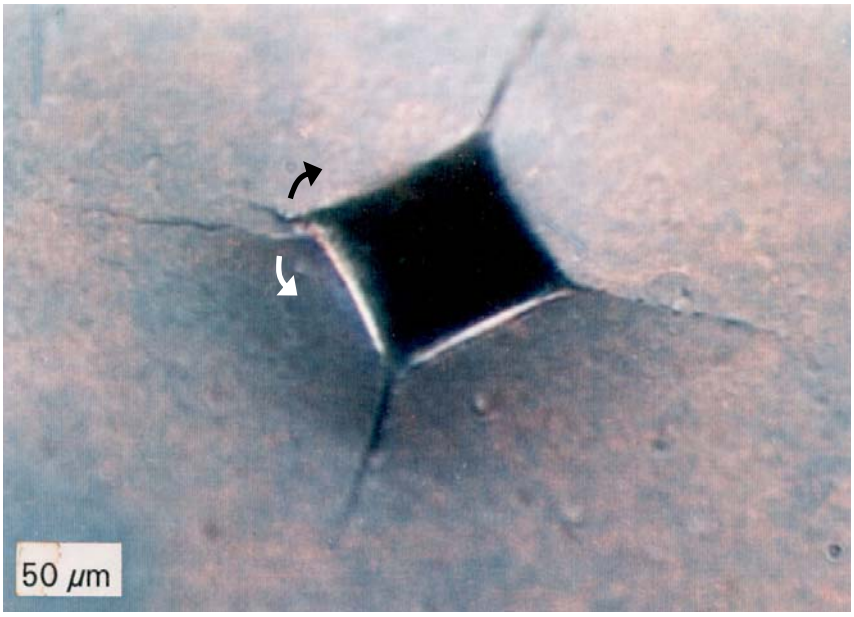

Figure 3 Oblique view of a 98 N (10 kgf) indentation revealing the significant uplift and curvature of the indentation sides. This image was made by tilting the specimen under a stereoptical microscope which has a much greater depth of field than a compound optical microscope. Some of the cracks seem to form where the indentation tips are peeled apart (arrows). Uplift along the sides of the indentation causes the tip sides to spread apart. The uplift also creates imaging problems in a compound optical microscope since, if the indentation tips are in focus, the crack tips and the curved indentation sides are out of plane and possibly out of focus. The tip peeling also contributes to irregularities in the indentation tip shapes.

[b] The microscopes attached to hardness testing machines may be adequate for routine size measurements of indentation sizes, but they are woefully inadequate for discerning and measuring the cracks.

eyepieces and slightly shifting the interference contrast polarizing filter. The view was also focused and defocussed. The crack tips were seen to shift slightly whereas the other microstructural features on the polished did not shift. Once a tip location was determined, then a marker-measurement line was put onto the computer monitor image at that precise location.

The best viewing conditions for the diagonal sizes (2a) was with bright field illumination, a green filter, a 40 X objective, and an auxiliary internal 1.6 X magnifier (Fig. 2a). A 100 X objective would have made the indentations appear larger on the computer monitor, but there was less contrast at the indentation corners and assessment of the tip location was more difficult than with the 40 X objective. Both diagonals were measured on every indentation. The lengths usually differed by only a few tenths of a micrometer. The maximum difference was less than 1 μm. Only indentations with four straight primary cracks were accepted. Indentations with badly split or forked cracks or with cracks coming from the sides of the indentation were rejected. Nearly all the indentations were acceptable.

SRM 2100 silicon nitride is not susceptible to slow crack growth at room temperature. K_{Ic} measurements made with the rigorous test methods in inert and ordinary atmospheric conditions produced the same results. Nevertheless, five 98 N indentations in the present study were made through a drop of silicon oil. A cover slip microscope slide was then placed over the drop to flatten it and the indentation taken to the microscope and measured. These indentation sizes were the same as those made under normal laboratory conditions. The crack lengths were 13 μm shorter (4.3 %) out of an average length of 298 μm. This is usually taken to be evidence of slow crack growth in the air environment, but in this case the difference was simply due to the greater difficulty of seeing the crack length when viewing through the oil. It is also possible that the oil acted as a lubricant and affected the indentation process.

Five 98 N indentations made in ordinary laboratory environment were remeasured after 17 days to see whether the crack lengths (2c) had increased. Surprisingly, they had all shortened by 3 μm to 14 μm and, on average, 8.7 μm (2.7 %). The digitally recorded images were reexamined at high power and the tip locations noted with respect to nearby tiny microstructural features that served as convenient location references. The crack lengths indeed did get shorter. This is the opposite of what would be expected if slow crack growth were active. It could be that the shortening is real or it was an optical effect analogous to stress free sharp cracks in glass plates seeming to healing if left undisturbed. It could also be evidence that the indentation residual stresses changed or relaxed.

In summary, a conventional hardness machine was used to make the indentations. A conventional research reflected light compound microscope with digital camera was used to measure the indentations and the cracks.

RESULTS

Table I lists the results. The SRM certified value is listed as K_{Ic} and the other estimates are shown as $K_{ifr.}$ For equations 2 and 10, the H or HV at the particular indentation load was used in the calculation. Only the 49 N and 98 N indentations had c/a ratios of 2.5 or greater. Figure 4 shows both K_{ifr} and the ratio $P/c^{1.5}$ versus indentation load.

None of the K_{ifr} outcomes match the SRM 2100 certified value. Figure 4 shows that the apparent fracture toughness became load independent at 49 N and above, corresponding to c/a ratios of 2.5 and larger. The closest outcomes were from the JIS R1607 equation 10, but these were still 5 % too low. Results from Niihara's equation 9 as used in ASTM F2094 were 21 % too high. Those from the Anstis et al. equation 2 were 19 % too low.

Table I. Vickers indentation fracture resistance results. Uncertainties are one standard deviation unless otherwise noted.

Indentation Load (N)	Average Diagonal half length a (μm)	Average Crack half length c (μm)	SRM 2100* Certified K_{Ic} (MPa√m)	ACLM** K_{ifr} eq. 2 (MPa√m)	Niihara*** K_{ifr} eq. 9 (MPa√m)	JIS R 1607 K_{ifr} eq. 10 (MPa√m)
9.8	16.2 ± 0.3	33.0 ± 1.4	4.57	3.44 ± 0.20	5.14 ± 0.30	4.02 ± 0.23
19.6	23.5 ± 0.1	52.0 ± 1.0	4.57	3.56 ± 0.11	5.29 ± 0.16	4.16 ± 0.13
49	37.5 ± 0.2	94.0 ± 1.5	4.57	3.69 ± 0.09	5.47 ± 0.13	4.31 ± 0.10
98	53.3 ± 0.3	148.9 ± 1.9	4.57	3.72 ± 0.08	5.51 ± 0.11	4.34 ± 0.09

* The uncertainty of SRM 2100 is ± 0.23 MPa√m for a single test outcome at the 95% confidence level. It is 0.11 MPa√m for the average of 5 test outcomes at the 95% confidence level.

** Anstis, Chantikul, Lawn, and Marshall, ref. 15

*** Niihara's equation with a constant value of 10.4 as per ASTM F 2094, ref. 10.

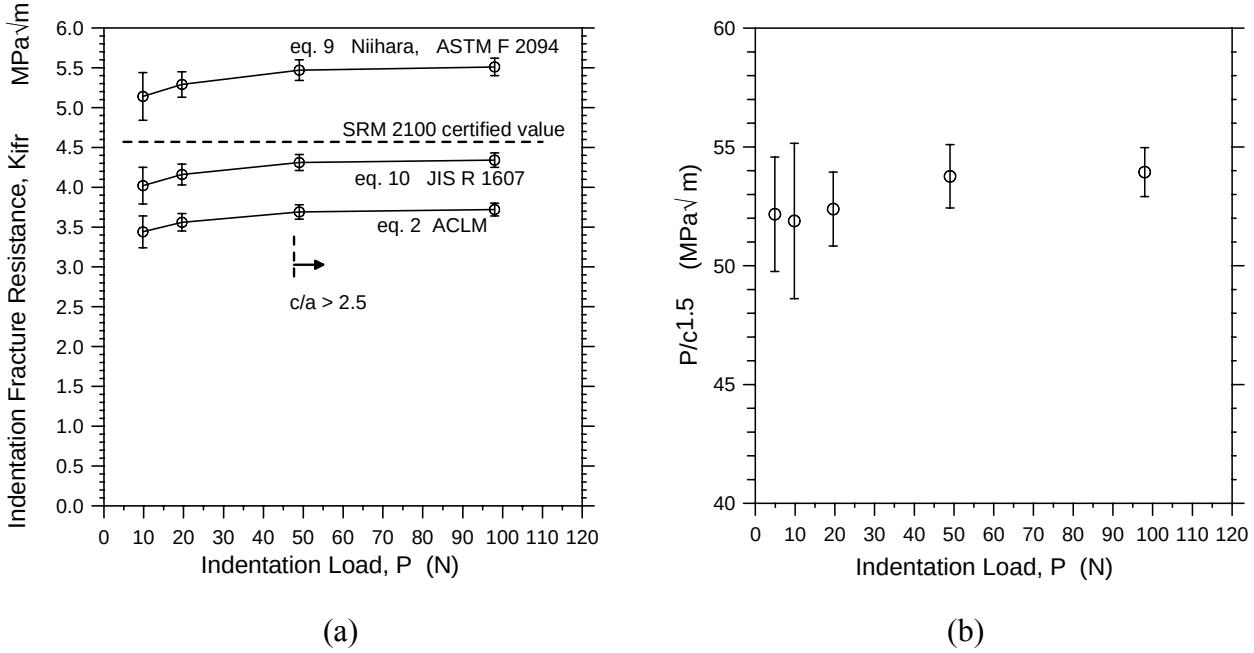

Figure 4 K_{ifr}, (a) and $P/c^{1.5}$, (b) versus indentation load.

The crack lengths in one direction (i.e., horizontally as viewed in Figure 2) were usually (but not always) longer than the cracks in the orthogonal direction. The differences were 6.0 μm, 5.0 μm, 22.7 μm, and 36 μm at the 9.8 N, 19.6 N, 49 N and 98 N loads, respectively. This is probably due to a fracture toughness anisotropy in this hot-pressed material.[7,9] The longer cracks ran in the hot-pressing plane of the original plate. The hot-pressing plane, which is perpendicular to the axis of pressure application during hot-pressing, is weaker due to a small preferred orientation of the slightly elongated beta silicon nitride grains in the material. There is little data on the fracture toughness of the material in this orientation and the NIST SRM 2100 is certified

only for planes parallel to the hot-pressing direction. These planes are perpendicular to the hot pressing plane. If the shorter crack lengths were used in the equations above, K_{ifr} increases by between 0.1 MPa√m to 0.35 MPa√m. The value from the JIS equation would be in good agreement with the certified value, but is probably fortuitous as discussed below. Additional experiments are planned with a new SRM 2100 broken bar that will be mounted with a 3 mm x 25 mm face for polishing in order to eliminate the anisotropy. Some experiments with a 196 N (20 kgf) indentation load will also be done.

DISCUSSION

In principle, the above results could be used to obtain another new calibration constant or even a new indentation fracture (IF) resistance formula, but that would be unwise as discussed below. Even if the results could be made to match the SRM 2100 certified value, it is doubtful whether the revised formula would be accurate for other materials.

The literature shows that the method frequently produces inaccurate results. Sometimes a plausible estimate of fracture toughness is obtained, but just as often, an incorrect estimate is obtained. As an example, in the 1980s the procedure was used to obtain an toughness of 3.8 MPa√m for a commercial sintered silicon carbide,[c,23] whereas the actual fracture toughness was 2.5 MPa√m to 2.9 MPa√m depending upon the batch.[1,24,25] Sometimes the method has been misused. For example, inflated K_{ifr} numbers can be obtained by using a low power microscope that cannot measure the full crack length. Often "custom fitted" or material-specific calibration constants are used.[d] Even if one settled on one equation, the calculated fracture toughness often depends upon the indentation load even for materials with a flat R-curve.

The large variability in results should not be surprising. The end of the 1981 Anstis et al. Part 1 paper[15] has a telling comment:

> "The combined random and systematic scatter in data suggests than an accuracy of better than 30 to 40 % should be attainable, at least for those materials which are well behaved in their indentation response."

This candid and perceptive warning has been ignored. There also is some room for doubt as to what constitutes a well-behaved material. Thirty percent may not seem too serious, but it is the difference in one laboratory reporting 3.1 MPa√m and another 5.9 MPa√m for a material with an actual fracture toughness of 4.5 MPa√m. Indeed, variability of IF data by a factor of two are common in interlaboratory studies as will be discussed below. The problems with the Vickers IF test are considered in turn.

A fundamental problem is that the equations are all suspect. Shortcomings in the model have prompted many authors to try to refine the model and adjust the equations to deal with data discrepancies. At present, there are probably more than forty variations of the fracture toughness – crack length equation in existence and none have been entirely satisfactory. Some are optimized for particular materials or indentation conditions. Li, Ghosh, Kobayashi, and Bradt[24] compiled and analyzed twelve variants. Ponton and Rawlings tabulated nineteen in their 1989 review papers.[26,27] For example, some have felt that the residual indentation plastic zone

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[c] Sintered alpha SiC, Hexoloy SA, Carborundum, Niagara Falls, NY. This material has no boundary phase and fractures transgranularly. It has constant fracture toughness and a flat R-curve.

[d] On several occasions, users have even refused to divulge a calibration constant to the author, saying that the formula and calibration constant were proprietary.

does not exert a constant force on the crack as is assumed, but instead acts as a wedge or alternatively as a compressed spring such that force decreases with crack extension.[28] Bleise and Steinbrech[29] pointed out that the residual stress parameter χ is not constant. It varied as much as 40 % with indentation load for a coarse-grained alumina. 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 which have created much confusion. The variants have different powers for the E, H, P and c terms. For a particular set of test data, they produce fracture toughness values that usually differ by as much as a factor of two or more.[26,27] Ponton and Rawlings[26] showed some results that varied by a factor of four for the same input data. None of the equations have been successful for a variety of materials.

All the equations have one weak point in common: an empirical calibration constant. These are inaccurate, imprecise, and vary with material. The Anstis et al.[15] empirical calibration constant of 0.016 in equation 2 was selected as the best compromise value to match double cantilever beam data for eight ceramics and glasses. As noted previously, there was considerable uncertainty ($\pm$ .004) in this value. The factor rolls up several geometry terms and aspects of the elastic-plastic damage zone around the indention. It is not likely that all brittle materials deform and fracture underneath an indentation in a self-similar manner as is assumed. For example, a covalently-bonded, hard ceramic (e.g., silicon carbide) deforms and fractures very differently than an ionic cubic ceramic (e.g., magnesium oxide), or a soda lime silica glass. Hence, it should not be surprising that the quest for the universal indentation fracture equation or calibration constant has been in vain.

The cracks often are not the idealized semicircular median cracks that are assumed in the models. The method relies solely on examining the surface traces of the crack. Few bother to examine the nature of the damage and cracking beneath the surface. The cracks are very complex and rarely are the idealized simple half penny shaped cracks commonly depicted in schematics. The crack system is actually a three-dimensional network of intersecting and interacting cracks (e.g., Hagan and Swain[30]). There are forked cracks, lateral cracks, and a network of shear microcracks underneath the indentation. Some cracks do not penetrate far below the surface and are shallow Palmqvist cracks. Revised formulae (with different crack length – load dependencies) have been developed for such cracks, but these alternative analyses do not remedy the underlying problem that the indentation crack is not a controlled single crack.

Median cracks and the plastic damage zones don't always form in accordance with the assumed models. The mechanics of crack formation were based on observations of crack initiation in a few model transparent materials such as soda lime silica. Subsequent work with broad ranges of glasses and transparent single crystals by Cook and Pharr[31] and many silicate and non-silicate glasses by Wilantewicz[32] show the crack formation sequence to be far more complicated than originally thought. Some cracks pop in on loading, some on unloading. Cook and Pharr said:[31]

> "Perhaps the most striking feature of our results is the lack of any general cracking sequence… We note that the cracking sequence in the normal glasses is in no way "typical" for brittle materials. In this context, previous choices of soda-lime glass as a prototype brittle material for the study of indentation cracking are questionable….."

In their study of the fracture mechanics and crack growth in glass, Michalske and Collins[33] observed that the indentations carry their own local stress field, but that "the indentation residual stress is known to decay with time, so that the χ value may be substantially reduced in the

interval between indentation and testing (≈ 10 min) and the constant force-spring assumption may be in error." They concluded:

> "The experimental findings … show no agreement with indentation fracture mechanics models."

An obvious but infrequently mentioned issue is that the final crack size measured is formed by a crack that decelerates and then stops. Most fracture toughness tests for K_{Ic} feature a crack that is loaded to a critical condition which leads to unstable rapid crack extension.

A practical problem is that it is difficult to measure the crack lengths on the surface. Any crack length uncertainty or error is magnified when K_{ifr} is calculated since the latter depends upon the crack length raised to the –1.5 power. Metrology issues in obtaining accurate diagonal size measurements for hardness are nontrivial, but the difficulties in measuring even tinier hairline cracks are often glossed over. Between-laboratory consistency is poor due to variations arising from microscopy limitations as well as operator experience or subjectivity. A 1988 Versailles Advanced Materials and Standards (VAMAS) international hardness round robin with nineteen laboratories[34] showed the between-laboratory variability in crack length measurements exceeded 20 μm for alumina. This difference was significant and a major source of the calculated fracture toughness scatter. Some laboratories even refused to do the measurements. A different VAMAS round robin on fracture toughness by three methods on silicon nitride and a fine-grained zirconia-alumina composite confirmed that estimates of the final crack-tip position were equipment sensitive and highly subjective.[35,36] Eighteen laboratories participated. The between-laboratory results varied by almost a factor of two for the fine-grained zirconia-alumina composite. It was concluded that:[36]

> "The IF results were disappointing primarily because of the high scatter and failure to obtain consistent interlaboratory results. The strong dependence of the computed fracture toughness upon the crack length and the difficulty in measuring such combined to cause high scatter."

Round robin participants stated:

> "Testing and interpretation is difficult." "Measurement of the crack tip is very subjective." "The method is not reliable."

It is commonly believed that as much as 20 μm can be added to an optical crack length measurement if the crack is measured with a SEM. In the second round robin, one participant reported a 10 μm difference between their optical and SEM readings.[36] The importance of these variations depends upon the size of the indentation and the cracks, but the errors were significant. Several participants observed that crack lengths were highly dependent on the mode of viewing. All laboratories said there was considerable interpretation as to where the exact crack tip was and there was difficulty measuring this point. Most agreed that the optics furnished with their microhardness machines were woefully inadequate for measuring the crack lengths. Most laboratories had to use more powerful microscopes. Similar findings came from an eighteen laboratory European Structural Integrity Society (ESIS) round robin in 1995:[37]

> "The results from the IF method are less accurate and exhibit more scatter compared to some other methods since length measurements of small cracks are required. In addition, the

results of the IF method are sensitive to subcritical crack growth. The method ….. cannot be used to compare fracture toughness values between laboratories."

Some of the photos of Vickers indentations and cracks in the ESIS study point out another problem: many indentations had multiple cracks and excessive shattering around the indentation.

Although the NIST SRM 2100 is not susceptible to slow crack growth under normal laboratory conditions, many other materials are susceptible. Environmentally-assisted slow crack growth can cause cracks to grow appreciably in the seconds and minutes after making the indentation.[15] Final measured crack size then reflects both the initial fracture resistance and subsequent stable extension due to slow crack growth. The fracture toughness will be underestimated. This is especially of concern for glasses, ceramics with glassy boundary phases, or oxide ceramics. Precautions may be taken such as indenting through silicon oil to retard the slow crack growth, but even this is not foolproof and indentations crack lengths must be measured immediately (within ~2 minutes) after the indentations are made.[15]

If there are so many problems, why do people still use the method? There are several reasons. Expediency is the most common. The more rigorous methods do require more effort and many users are looking for a short cut. The simplicity of the method also has appeal. There are times when there simply is not enough material available to do a conventional test.

The numerous papers, presentations, and books with data by the method have given the impression that the method is favored for most applications. The elaborate foundation papers in the 1970s and 1980s have lent more credence to the method than is warranted. The reality is that those who are familiar with the method do recognize its shortcomings. The problems are glossed over by contending that the procedure is simply a research tool to study material behavior. Sometimes it is said that results are merely needed to rank material and the exact values are unimportant. This is a weak and overused argument that can cause long term harm.[e] The Vickers indentation fracture method may be a useful research tool, but users cross a dangerous threshold when they place too much value in the veracity of the data, especially if it is to be used for design or material specifications purposes.

Newcomers to the field are unaware of all the shortcomings. They see the large volume of published indentation fracture results and assume the method must be generally accepted. The simplicity of the method is attractive and it seems ideal for a quick estimate of fracture toughness especially if insufficient material is available for traditional fracture toughness specimens. After they have done the IF experiments, conscientious newcomers check on whether the results are reasonable and they scan the literature for comparisons. They are surprised and disappointed at the variability they find. Often they follow up by doing a few supplemental tests on a convenient "quasi reference" material such as a dense fine-grained alumina, but even here there are conflicting results in the literature. Sometimes the newcomer will try one of the alternative equations and they will obtain different numbers. By now the newcomer is confused and is not sure what to believe. Less conscientious workers have simply published the numbers without worrying about their veracity.

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[e] The author heard the same argument 25 years ago in the context of flexural strength testing. The then state of the art of testing was very poor. The "data for ranking purposes" argument was used to justify sloppy and faulty test procedures. Modest test method reforms and standardization led to quantum improvements in data quality, lower costs, and better credibility. With a few exceptions, the state of the art of flexural strength testing now is very good.

Although there is widespread open skepticism about this procedure, only a few have been candid in print. A number of warnings have been raised by many as cited previously, but these voices have been overwhelmed by the din of uncritical publications. It is worth reviewing some of the other warnings. Paddon and Morrell were quite frank:[38]

> "Various tests have been devised for determining fracture toughness of brittle materials. With the exception of the somewhat *discredited* [f] indentation crack length measurement and NPL's edge-toughness test currently under development, all require the test-piece to have a well developed crack or machined notch to act as a defined defect of known geometry with which to commence the measurement."

In one memorable contribution, Li, Ghosh, Kobayashi, and Bradt[39] wrote:

> "Because of the ease of application of the method to small specimens, many researchers continue to utilize the Vickers microfracture technique, frequently without seriously questioning either the method itself or the toughness results. ……
> …. the existing equations, as previously "calibrated," simply do not yield the correct values of the fracture toughness. All previously published fracture toughness results using the Vickers indentation microfracture method and applying any of the previously summarized equations, particularly when crack lengths and hardness are measured simultaneously, can be accepted only with considerable reservation. It is probably not unreasonable to *simply reject the majority of those fracture toughness measurements as incorrect*."[f]

and then later:

> "Although elastic plastic models have been applied to model the median crack system, the large-scale plastic strain which exists for metals is not present for ceramics. Thus, the model is a convenient, although perhaps a somewhat unrealistic representation of the genesis of the indentation crack system in ceramics. Aside from the fact that the indentation technique frequently does not yield reasonable toughness values utilizing many of the equations which have been proposed it has other drawbacks. These drawbacks include asymmetrical crack patterns, peculiar "R-curve" behavior, questionable accuracy, and widely differing results when applied to different ceramic materials."

Even Fujii and Nose, two primary authors of JIS R 1607, had strong doubts:[40]

> "It is troublesome, however, that the "K_{Ic}" value differs with the proposed formula, due to the difference in the models, in the reference materials, and in the reference test methods by which the K_{Ic} values were measured to fit those parameters ……"

Although they stated that the K_{ifr} values correlated reasonably well with some SEPB results, there were lingering doubts:

> "As consequence, the IF method was also included in the original form of a JIS, where the "K_{Ic}" value has been denoted by K_c to be distinguished from the K_{Ic} evaluated by the SEPB

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[f] Emphasis added.

method. It should be noted, however, that the IF method is useless to evaluate K_c of some materials, where median/radial cracks are obscure, and there is no assurance that the method can always be applied to newly-developed materials."

One of the early advocates has even had second thoughts. Over a decade after his 1976 paper[13] precipitated widespread usage of the procedure, Professor Evans said in his 1990 Orton Memorial lecture at the Annual Meeting of the American Ceramic Society:[41]

"Various indentation methods allowed tests on small specimens and thus, permitted rapid probing of the 'damage tolerance' of many different ceramics. …… Many of the indentation methods are only approximate and do not provide the quality of fracture resistance data needed to rigorously relate toughness to microstructure."

He added

"The surface flaw methods, introduced by Petrovic and Jacobson, seem to be the most precise, provided that residual stresses are eliminated by polishing out the plastic zone."

The latter approach is a refined indentation method entailing creation of a single, controlled semielliptical crack by Knoop indentation in a flexure strength specimen. The residual stress damage zone is removed by polishing. This fracture mechanics method is now known as the surface crack in flexure (SCF) method and has been standardized in ASTM,[1] CEN,[3] and ISO.[5]

The ASTM task groups involved with fracture toughness standardization never seriously considered Vickers IF as a candidate standard (e.g., ASTM Committees E-08, Fracture and Fatigue and C-28, Advanced Ceramics).[42,43] The fracture mechanics community has strong reservations about the method.

In summary, while the Vickers indentation crack length method is commonly used for research purposes, experience belies the method's suitability as a reliable method for producing accurate results for the fracture toughness, K_{Ic}. Sometimes it gives credible results, but just as often it does not. One really cannot be sure that one even gets a "ballpark number." Many of the conclusions drawn from the data are unsound. For example, faulty fracture toughness data generated by the Vickers IF method caused one commercial silicon nitride material to be misclassified since it failed to meet a specified fracture toughness level in a formal material specification. Fracture toughness data from technically rigorous methods showed the material did in fact meet the specification.[i]

Some contend that the rigorous fracture toughness tests require too large a test piece. There is some truth to this complaint, but progress had been made in miniaturizing some of the proper fracture toughness tests. Small bend bars (20 mm long) may be tested by the SEPB, SCF, or CNB methods on very short (16 mm to 20 mm) bend fixtures. These methods have the basic ingredients of a bona fide K_{Ic} fracture toughness test: a single well-shaped precrack, a good loading configuration, and an accurate stress intensity factor solution.

[i] A commercial hot-isopressed silicon nitride that was characterized by a major European ball bearing manufacturer. Vickers IF data produced a K_{ifr} of 4.1 or 4.2 MPa$\sqrt{m}$ causing the material to fall outside a level III classification which required a minimum of 5.0 MPa$\sqrt{m}$. The K_{ifr} values were much too low. SCF experiments by the author in accordance with ASTM C 1421 gave an average K_{Ic} of 5.4 MPa$\sqrt{m}$ ± 0.24 MPa$\sqrt{m}$.

A common disclaimer is that the Vickers indentation fracture data are being collected for rough, comparative purposes. This may be so, but when the data is published, it often appears in tables as "fracture toughness, K_{Ic}." It would be best to simply report the data as "indentation fracture resistance, K_{ifr}" which may or may not approximate the fracture toughness K_{Ic}.

Is the indentation fracture resistance K_{ifr} a useful property in its own right? Perhaps it is. It measures the resistance to crack extension from a particular type (Vickers) of indentation. The indentation has a lot of localized damage, but so do localized events associated with wear or machining processes. Keep in mind however, that few contact damage sites created in service or by machining or wear are created in as controlled a fashion as a Vickers indentation.

CONCLUSIONS

The Vickers indentation crack length method has numerous drawbacks. A universally accepted equation does not exist and probably never will, since different materials have dramatically different deformation and cracking characteristics. The method does not produce accurate and precise fracture toughness K_{Ic} values. The results are only approximations and can easily be as much as 50 % to 100 % in error. Vickers indentation crack length data are better described as: "indentation fracture resistance, K_{ifr}."

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