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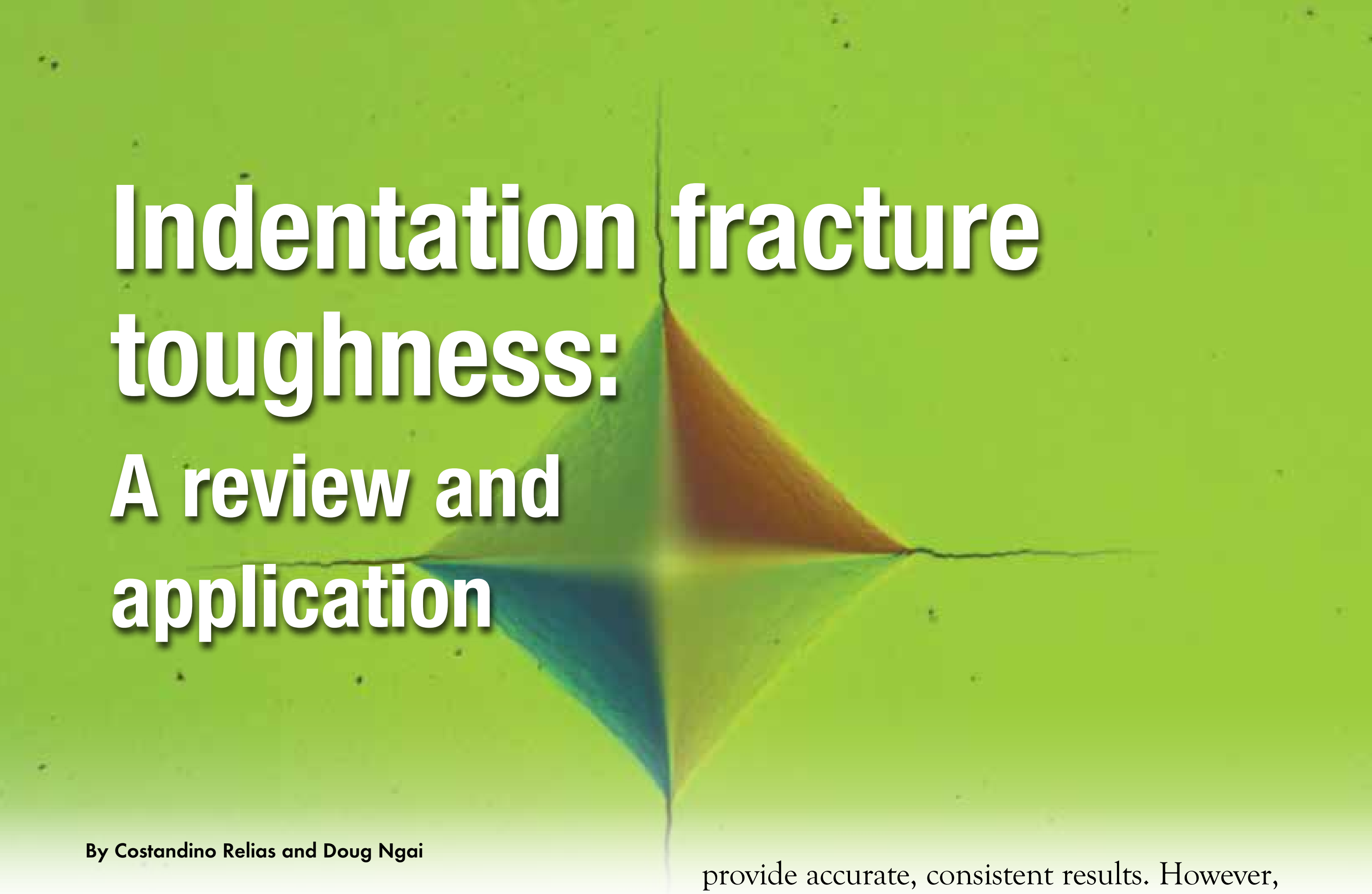
New advances in sintering | Indentation fracture toughness testing | Keramos' deep history and bright future



Indentation fracture toughness:

A review and application

By Costandino Relias and Doug Ngai

Vickers indentation eliminates the need for standardized samples to determine fracture toughness of brittle materials. The key is to select the correct system of equations to calculate fracture toughness from crack morphology.

Fracture toughness (K_{Ic}) is an intrinsic material property that defines how well a material resists fracture. The standardized testing methods for K_{Ic} involve creating a small crack in a fracture toughness test specimen and propagating the crack under an applied load. Examples of standardized methods for ceramics and brittle materials include: the chevron-notched beam (CNB), single-edge precracked beam (SEPB), and the surface crack in flexure (SCF) methods.[1] These methods are described in ASTM C1421, and when performed in accordance with standards, they provide accurate, consistent results. However, these tests must have a designated specimen geometry and use with specific test equipment. In contrast, the indentation fracture toughness (IFT), K_{Ic}, test method does not have significant restraint to specimen size or geometry; the specimen, typically, only requires a scratch-free, 1μm surface finish. The ease of testing and low financial cost of the indentation fracture method compared to standardized methods makes it a preferred choice for estimating fracture toughness of brittle materials.[1]

The indentation fracture toughness testing uses cracks emanating from Vickers hardness indents to estimate the material's K_{Ic}. These cracks are formed when high load Vickers indentation is applied to the test specimen. The ideal crack profile has one crack protruding from each corner of the indent, as shown in Figure 1. If there are multiple cracks and/or uneven crack lengths, then the indent would not be suitable for K_{Ic} estimation. An example of a poor crack profile is shown in Figure 2.

When an ideal indent is created, a multitude of equations may be considered to calculate fracture toughness from the crack length information. These equations originate from different experimental studies and will be discussed later. Unfortunately, no single equation can measure fracture tough-

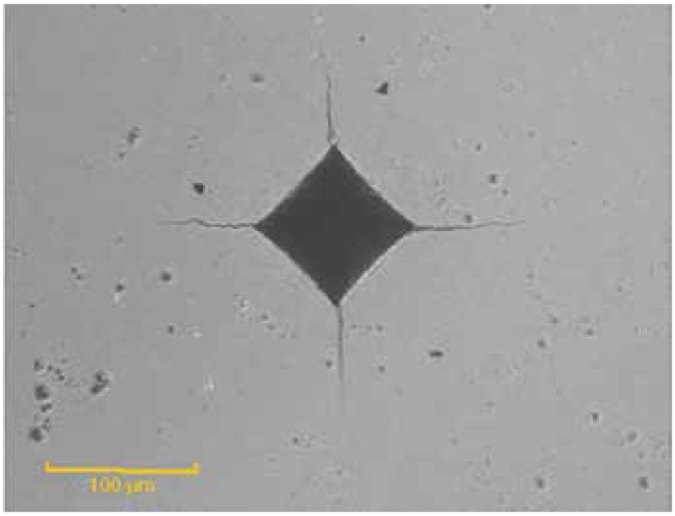


Figure 1. A 98N Vickers indent in Si_3N_4 with ideal cracks.

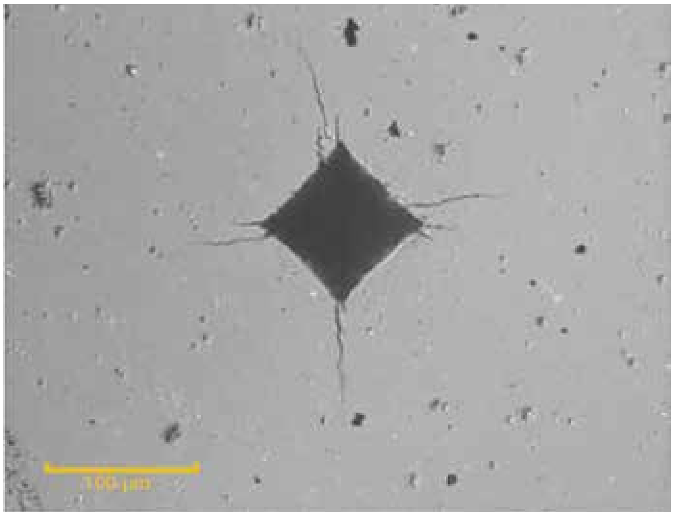


Figure 2. A 98N Vickers indent in Si_3N_4 with irregular cracking not suitable for K_{1c} calculation.

ness accurately for a variety of different materials; thus, a standardized method of performing indentation fracture toughness testing has not been established.[1,2,3]

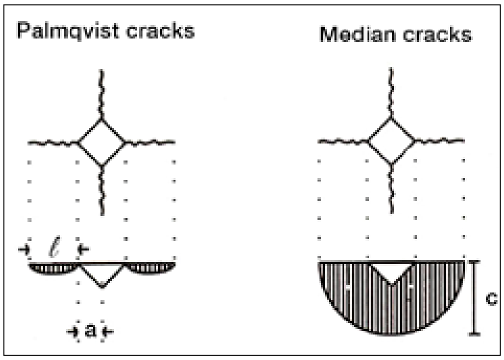


Figure 3. Crack profile comparison.[4]

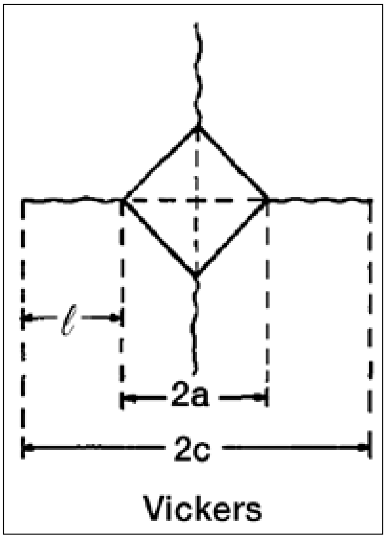


Figure 4. Defined lengths of Vickers indent and cracks.[6]

Crack systems and fracture toughness equations

With Vickers indentation fracture toughness testing, two possible crack systems may be considered. These two crack systems are Palmqvist cracks and Median cracks. The type of crack formed depends on the material and indentation load, with the Palmqvist cracks forming at low loads or in high fracture toughness materials while the Median cracks form under opposite conditions.[4,5] The Palmqvist crack system consists of four cracks that initiate at the corners of the Vickers indent and stay close to the material's surface. In contrast, the Median crack system has a half-penny profile which contains two cracks that extend under the indent.[5] These two crack systems are depicted in Figure 3.

Because these crack systems appear the same from the surface, the only way to verify the actual crack system is to examine the cross section of the indent. This is not feasible when performing indentation fracture toughness testing, so the *c/a* ratio was developed to determine which crack system is formed with each indent. The value *c* is the radius of the half-penny crack, *a* is the indent half-diagonal and is the average crack length. These values are used in the indentation fracture toughness equations and correspond to the lengths shown in Figure 4. According to Niihara, when $c/a > 2.5$ the crack is assumed to be Median and below 2.5 it will be Palmqvist.[7] Other studies have shown that there is a transition period where the crack has a possibility of being both.[5] The general rule is that if $c/a > 2.5$ then the crack system is most likely to be Median; while, if $c/a < 2$, then it is most likely Palmqvist. If it is in between, it could be either depending on the material.

Determining the crack system is important for indentation fracture toughness calculations, as it regulates which equation should be applied. The origin of many indentation fracture toughness equations is based on fracture mechanics analysis and fitting of experimental data. The formation of Median cracks is considered to be equivalent to a center loaded crack at the indent, while Palmqvist cracks are equivalent to a semi-infinite crack loaded at each indent corner.[7] This difference in modeling results in the variance in the equation form between the crack systems. Below are the most widely used indentation fracture toughness equations for each crack system.

Palmqvist crack system equations

Shetty equation[8] $K_{Ic} = 0.04285\ 1/(1-\nu^2)\ (HP/l)^{1/2}$

Niihara equation[7] $K_{Ic} = 0.0123\ (E/H)^{2/5}\ (HP/l)^{1/2}$

Median crack system equations

Anstis equation[9] $K_{Ic} = 0.016\ (E/H)^{1/2}\ (P/c^{3/2})$

Niihara equation[7] $K_{Ic} = 0.0309\ (E/H)^{2/5}\ (P/c^{3/2})$

Miyoshi equation[10] $K_{Ic} = 0.018\ (E/H)^{1/2}\ (P/c^{3/2})$

E = Young's Modulus (GPa)
H = Hardness (GPa)
P = Indent force (N)
l = Average crack length (10^{-3} m)
c = Average length (10^{-4} m)
ν = Poisson's Ratio

The Anstis and Miyoshi equations use the same basic form, based off a center loaded half-penny crack.[9] The difference between these two equations is its respective constant, which is determined by fitting the equation with experimental data. Anstis used various ceramic materials to fit to the equation, while the Miyoshi equation was mainly focused on silicon nitride. The Niihara equations have a slightly different form because they are based on curve fitted behavior, not crack models. Niihara has two equations because they determined from the fitting of experimental data sets for Palmqvist and Median cracks separately.[7] The other Palmqvist crack equation used to estimate fracture toughness is the Shetty equation. This equation uses Poisson's ratio instead of Young's Modulus and is based off a wedge loaded two dimensional through crack.[8]

Indentation fracture toughness application

The concern with using indentation fracture toughness to estimate K_{Ic} is that for every situation and material, different equations need to be evaluated and considered. When performing indentation fracture toughness testing on a general ceramic material, Miyoshi's equation has shown to provide a rough correlation with actual fracture toughness values for most ceramics.[3] According to Miyazaki and Yoshizawa, Miyoshi's equation tends to underestimate fracture toughness while Niihara's equation for Median cracks tends to overestimate it.[3] The Miyoshi equation is for Median crack systems, so a sufficiently high load is required to ensure $c/a > 2.5$. If the $c/a > 2.5$ ratio is not attainable, a c/a ratio greater than 2 will be adequate for most scenarios.

Some materials provide more accurate indentation fracture toughness results and have a specified equation that is more applicable. For example, when testing Si_3N_4, using Miyoshi's equation shows good correlation with actual fracture toughness values and provides an accurate estimation.[1,3] Two other ceramic materials that have received notable attention are WC-Co and SiC. Multiple studies using indentation fracture toughness on WC-Co cemented carbides have concluded that Shetty's equation provided the best fit with actual fracture toughness values.[4,11] WC-Co cemented carbides are materials with a high fracture toughness, so the Palmqvist crack system forms in most scenarios. Therefore, K_{Ic} calculations using Median crack system equations will be inaccurate. SiC is another popular ceramic material tested significantly with the indentation fracture toughness method. In most cases, using the Miyoshi equation with SiC will result in an overestimation of fracture toughness and studies have shown that the Anstis equation provides the most accurate K_{Ic} value.[1,2,3] Figure 5 is a guide to select the proper equation based on material.

Comparative testing is another way to utilize the indentation fracture toughness method. The indentation testing is a fast and convenient method to compare various samples' fracture toughness. Especially those specimens that cannot be measured by other methods. This comparative testing is best done with the same type of material and caution should be used when comparing different materials that have similar fracture toughness values. A good example of comparative testing is examining the fracture toughness of a material with different alloying elements or composition changes. This type of experimentation and assessment is shown in Soleimanpour, et al., for WC-Co cemented carbides.[11]

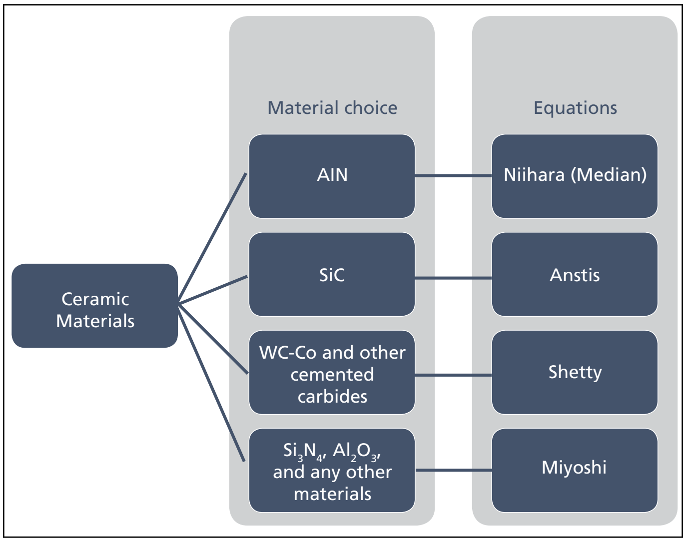


Figure 5. Diagram showing the equations that are suitable for the various variety of ceramic materials including: AlN,[3] SiC,[2,3] WC-Co,[4,11] Si_3N_4,[3] Al_2O_3,[3] and any others.

Silicon nitride indentation fracture toughness experimental tests

A 3M silicon nitride specimen with a reference fracture toughness value of 6 $MPa\cdot m^{-2}$ was used to demonstrate effectiveness of the indentation fracture toughness method and provides an example of how each equation relates to one another. A piece of Si_3N_4 material was sectioned, mounted, and polished in preparation for the testing. After specimen preparation, the indentation fracture toughness testing was performed using a Buehler VH3100 automated hardness tester, integrated with Diamet software, v1.7. Three loads were used for testing (29.4, 49, 98 N) and five tests were performed for each load.

Figure 6 and Table 1 show the indentation fracture toughness values calculated using each equation for Median and Palmqvist crack models. The Palmqvist crack equations show a load dependence of the fracture toughness values which implies that the Palmqvist model does not fit with Si_3N_4 material and the Palmqvist equations should not be used calculating Si_3N_4 IFT value. In contrast, the Median crack equations show no load dependence, and the fracture toughness values calculated by the Miyoshi equation are very close to the reference value of 6 $MPa\cdot m^{-2}$. These experimental results agree with the earlier conclusions that the Miyoshi equation is best for estimating indentation fracture toughness of silicon nitride.

Concluding remarks

The indentation fracture toughness method is a quick and simple assessment for estimating fracture toughness values. When the test specimen is too small and/or unique in size to meet standardized fracture toughness methods' specimen

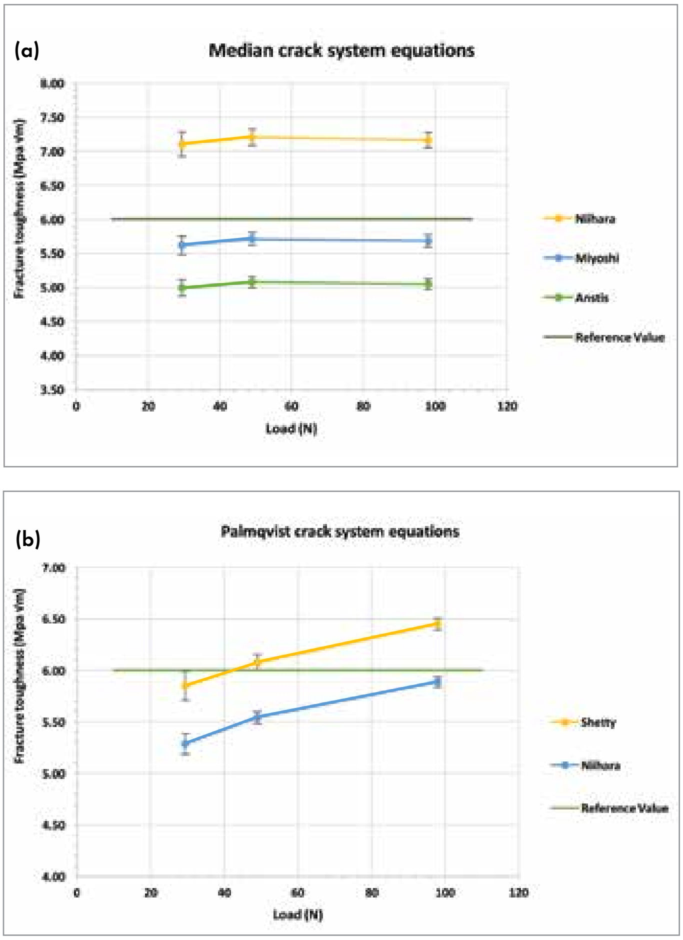


Figure 6. Indentation fracture toughness results of silicon nitride with a reference value of 6 MPa·m^{-2}. The error bars represent +/- 1 standard deviation. (a) Results from the median equations do not show load dependence for silicon nitride fracture toughness value. (b) The Palmqvist equations show load dependency for this material. These results determine that the Palmqvist crack equations should not be used for silicon nitride.

geometry, IFT is an option to estimate fracture toughness. Even though, the application of this method to assess fracture toughness varies with the selection of equations for different materials, it is still a useful tool for certain materials and applications. The Miyoshi equation is an ideal choice for a variety of ceramic materials and has been shown to give good agreement with reference fracture toughness values for silicon nitride.

Indentation fracture toughness is a useful tool for fracture toughness estimation. With a good understanding of the various equations, matching them with the proper material can provide accurate results. More detailed information about how to perform indentation fracture toughness testing will be published in the Jan/Feb 2019 *ACerS Bulletin*.

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[12] Indentation Fracture Toughness Application Guide ■

Table 1. Indentation fracture toughness results of silicon nitride with a reference value of 6 MPa•m-2. The Miyoshi equation gave the most accurate fracture toughness values.

Indentation load (N)	Average diagonal half-length, α (µm)	Average crack length, l (µm)	Fracture toughness values				
			Antis (median)	Miyoshi (median)	Niihara (median)	Niihara (Palmqvist)	Shetty (Palmqvist)
29.4	30.59 ± 0.28	26.80 ± 0.95	4.99 ± 0.12	5.62 ± 0.14	7.10 ± 0.18	5.29 ± 0.10	5.85 ± 0.14
49	39.94 ± 0.27	40.44 ± 0.87	5.08 ± 0.08	5.71 ± 0.09	7.20 ± 0.12	5.54 ± 0.06	6.07 ± 0.08
98	56.53 ± 0.27	71.59 ± 1.30	5.05 ± 0.08	5.68 ± 0.09	7.16 ± 0.11	5.89 ± 0.05	6.45 ± 0.06