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Surface Crack in Flexure versus the Vickers Indentation Method for calculating Fracture Toughness in two veneering porcelains

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Abstract

Surface Crack in Flexure (SCF) is one of three accepted and internationally standardized methods for measuring fracture toughness (K_{IC}) of dental porcelains. There are more studies that use the Vickers indentation method (VI), despite it being criticized in the literature as being inaccurate.

Objectives: To compare the VI method against the standardized SCF ASTM C1421(2010) (ISO 18756: 2003) using two different metal-ceramic veneering porcelains.

Methods: Twenty SCF bar specimens were fabricated in accordance with ASTM C1421, (2010) using Vita VM13 (n=10) and Wieland REFLEX (n=10) ceramic. K_{IC} for each ceramic was calculated using the method prescribed in the standard. The same specimens used in the SCF groups were used for the VI method; five Vickers indentations were made on each specimen.
The indents were measured under a light microscope and K_{IC} calculated.

Results: K_{IC} measurements were statistically significantly higher in the VI method when compared to the SCF method ($p<0.05$) for both veneering porcelains. The mean K_{IC} (standard deviations) recorded for Wieland REFLEX were 0.75 (0.10) MPa $m^{1/2}$ using the VI method, and 0.65 (0.03) MPa $m^{1/2}$ using the SCF method, while Vita VM13 had slightly higher K_{IC} values at 0.93 (0.06) MPa $m^{1/2}$ for VI and 0.76 (0.04) MPa $m^{1/2}$ for SCF.

Significance: The VI method appears to overestimate the K_{IC} of the porcelains tested in this study when compared to the standardized SCF method. Caution must be taken when using indentation fracture toughness methods to report absolute fracture toughness values for dental porcelains and ceramics, and standardized methods should be used and adhered to.

Introduction

Fracture toughness (K_{IC}) is defined as the critical stress intensity factor, Mode I, of fracture. It is a measure of the resistance to crack propagation in brittle materials (Anusavice et al. 2012). It indicates the extent to which a material can resist rapid crack propagation, while providing insight as to how reliable and serviceable a ceramic restoration can be (Choi et al. 2011). Several techniques have been developed over the past few decades as a means of testing this innate material property. A Vickers indentation fracture test (VI) was first utilized in the 1970s (Evans and Charles 1976) and has become increasingly popular due to its expediency, convenience and low financial cost (Yoshimura 2005). Despite its popularity, the VI technique is criticized by various authors throughout the literature. Guazzato et al. (2004) have described the VI test as “not meeting fracture mechanics criteria”. Likewise, Quinn and Bradt (2007) recommend that the VI technique should no longer be used as a method of testing fracture toughness.

Surface Crack in Flexure is a standardized fracture toughness method, ASTM C1161-02c (2008), and is extensively used in engineering ceramics fields. The SCF method differs to the VI method in that K_{IC} is determined by fracturing a specimen that has a semi-elliptical surface pre crack initiated by a Knoop indentation. However, unlike the VI, residual stress fields are removed from the specimen by polishing and thus removing the indentation, after which the specimen is fractured using a four-point fixture. The SCF method was featured in the Versailles Advanced Materials and Standards (VAMAS) round robin in 1994, involving 22 laboratories evaluating the fracture toughness of two silicon nitrides and one yttria-stabilized zirconia. The technique demonstrated excellent reproducibility (coefficient of variation is 6.6 – 8.9%) and repeatability (coefficient of variation is 5.4– 7.7%) (Quinn et al. 1995, Quinn 2002).

Therefore, the purpose of this study was to compare the K_{IC} measurements of two veneering porcelains using two fracture toughness test methods, namely the SCF method and the VI method.

Materials and Methods

Twenty SCF bar specimens were fabricated using two different veneering materials (Table 1) in accordance with ASTM C1421 (2010). Both the SCF test as well as the VI test were performed on the same specimens allowing for a direct comparison of the results. All specimens were fired in an Austromat M ceramic furnace (DEKEMA Dental-Keramikofen GmbH, Germany) following the respective manufacturers’ instructions with the exception of a slow cooling protocol of nine minutes. This was to minimize residual stress within the specimens.

The specimens were planed under irrigation using 400 grit silicon carbide abrasive paper (Struers Inc, OH, USA.) on a rotary polishing machine (Struers Inc.), ASTM C1161-

Table 1. Porcelain porcelains tested for fracture toughness in both testing methods *According to manufacturers' information

Veneering Porcelain	Type	Composition*	Manufacturer	CTE [K-1]	N
Wieland REFLEX	Feldspathic	Nano-leucite containing porcelain	Wieland Dental & Technik GmbH & Co KG, Pforzheim, Germany	13.1 x 10^{-6}	10
Vita VM13	Feldspathic	Fine containing leucite porcelain	VITA Zahnfabrik, H. Rauter GmbH & Co KG, Bad Säckingen, Germany	13.6 x 10^{-6}	10

02c (2008). The surface that was to be used for the VI test method was further polished with 2000 grit silicon carbide abrasive paper until the final dimensions were achieved. All specimens were annealed after polishing, according to manufacturers' instructions.

Surface crack in flexure method

In order to eliminate laboratory result bias in the current study, the authors deemed it necessary to calibrate the laboratory setup as suggested by Quin and Bradt (2007). The setup calibration was initially performed using 5 silicon nitride beams of equal dimensions to the tested porcelain specimens. K_{IC} values from the calibration fell within the certified reference value of fracture toughness of 4.57 MPa $m^{1/2}$ ±0.11 MPa $m^{1/2}$ at a 95% confidence level (Quinn and Bradt 2007; ASTM C12421, 2010).

SCF indentation was performed in accordance with ASTM C1421-10, using a universal testing machine (Instron 3369, Instron Corp. IL, USA) with a 100N load cell and a Knoop indenter (ASTM C1421, 2010). The indentation load was set at 30N with a dwell time of 15 seconds. Indentations were viewed and measured using a stereoscopic zoom microscope (SMZ800, Nikon Corporation, Tokyo, Japan) to measure the long diagonal (d) of the Knoop indentation. The approximate depth of indents were calculated using the following formula:

Equation 1 $$h = d / 30$$

Where:
h = depth of Knoop indent
d = length of long diagonal for a Knoop indent

Indents were removed under irrigation using 400 grit silicon carbide abrasive paper and rotary polishing machine. The specimens were then mounted on a fully articulated four point bend jig (Model No. WTF-CF, Wyoming Test Fixtures Inc. UT, USA) and brought to failure with a 0.5mm/min load rate using the universal testing machine and a 500N load cell. The data were captured using Bluehill 2 software (version 2.3.359, Instron Corp.). Each fractured surface was examined using a scanning electron microscope (SEM) (JSM 6700 FESEM, JEOL; Japan) with the backscatter function used to improve depth of field and visualization of precracks. Crack pattern for each specimen was measured and K_{IC} was calculated using the following formulae:

For the deepest point of the precrack:
Equation 2

$$Y_a = \frac{[\sqrt{\pi} M H_2]}{\sqrt{Q}}$$

Where:

$$Q = Q(a/c) = 1 + 1.464[a/c]^{1.65}$$

and:

$$M = M(a/c, a/W)$$

$$= [1.13 - 0.09[a/c]] + \left[0.5 + \frac{0.89}{[0.2 + [a/c]]}\right][a/W]^2 + \left[0.5 - \frac{1}{[0.65 + [a/c]]} + 14[1 - a/c]^{24}\right][a/W]^4$$

$$H_2 = H_2(a/c, a/W) = 1 - [1.22 + 0.12[a/c]][a/W] + [0.55 - 1.05[a/c]^{0.75} + 0.47[a/c]^{1.5}][a/W]^2$$

Where:
W = the top to bottom dimension of the test specimen parallel to the crack length (depth).
a = the crack depth (m) as seen in figure 2.
c = the crack half width (m) as seen in figure 2.

For the point at surface:

Equation 3

$$Y_s = \frac{[\sqrt{\pi} M H_1 S]}{\sqrt{Q}}$$

\Where:

$$H_1 = H_1(a/c, a/W) = 1 - [0.34 + 0.11[a/c]][a/W]$$

$$S = S(a/c, a/W) = \left[1.1 + 0.35[a/W]^2\right]\sqrt{a/c}$$

To determine K_{IC}:

Equation 4

$$K_{ISC} = Y\left[\frac{3P_{\max}[S_o - S_i]10^{-6}}{2BW^2}\right]\sqrt{a}$$

Where:

K_{ISC} = the fracture toughness, sc method (MPa)
Y = the stress intensity factor coefficient (dimension-less)
$P_{\max}$ = the maximum force (N),
S_o = the outer span (m),
S_i = the inner span (m)

Vickers indentation fracture toughness method

After the SCF specimen measurements were completed under SEM, the specimens were retrieved and the test surfaces polished with 2000 grit silicon carbide abrasive paper to eliminate residual surface stress. These were sputter-coated with 10nm of Au-Pd using an Emitech K575X Peltier-cooled high resolution sputter coater (EM Technologies Ltd., Kent, UK). A universal testing machine (Instron) was used to produce indentations made with a Vickers hardness indenter (Shimadzu Corp., Kyoto, Japan) using a standard 136° pyramidal diamond indenter, at a load of 20 N with a dwell time of 15 seconds. Digital photographic images were taken immediately after each indentation using a digital camera (PowerShot A640, Canon, Tokyo, Japan) that was fixed onto a light microscope (Alphaphot-2 YS2, Nikon, Tokyo, Japan) to ensure minimal error of crack lengths due to continuing crack propagation in the presence of residual indentation stress and environmental moisture. Using Adobe Photoshop CS6 software (Adobe Systems Inc., San Jose, USA) each indentation was measured at a later date. K_{IC} was calculated in Microsoft Excel 2013 (Microsoft Corp., Redmond, Washington, USA) using the following formula:

Equation 5

$$K_{Ic} = k\left(\frac{E}{H}\right)^{0.5}\frac{P}{c^{3/2}}$$

Where:

K_c = Fracture Toughness MPa $m^{1/2}$
P = Load applied = 20 N
E = Elastic modulus (Pa)
c = Length of radial crack (m)
a = Indent length (m)
k = 0.016 constant
H = Hardness = P/A = P/Indent length Sq

Paired t-test statistical analysis was performed with SPSS software (IBM Corp., Armonk, NY, USA) with a significance level set at 95%. The mean coefficient of variation between the methods were determined using Excel 13 software. Bland Altman plots were produced using Excel 13 software (Microsoft) to describe agreement between two quantitative measurements including limits of agreement.

Table 2. Mean Fracture toughness (K_{IC}) values for the two ceramic types

Porcelain type	VI K_{IC} [MPa $m^{1/2}$] (sd)	SCF K_{IC} [MPa $m^{1/2}$] (sd)
Wieland REFLEX	0.75 (0.1)	0.65 (0.03)
Vita VM13	0.93 (0.06)	0.76 (0.04)

Results

Paired t-tests were performed to determine significant differences in K_{IC} between the two ceramic types (Table 2) as well as significant differences in the two fracture toughness test methods (Table 3). The mean K_{IC} (and standard deviations) recorded for Wieland REFLEX were 0.75 (0.10) MPa $m^{1/2}$ by the VI method, and 0.65 (0.03) by SCF, while Vita VM13 had slightly higher K_{IC} values at 0.93 (0.06) MPa $m^{1/2}$ for VI and 0.76 (0.04) MPa $m^{1/2}$ for SCF. K_{IC} was significantly higher (P=0.006 for Wieland REFLEX and P=0.000 for Vita VM13) in the

Table 3. Paired samples t-test (p<0.05) for comparing mean K_{IC}, measured by VI fracture toughness testing vs SCF fracture toughness testing.

Paired Samples Test									
		Paired Differences							
				95% Confidence Interval of the Difference					
	Mean	Std. Deviation	Std. Error Mean	Lower	Upper		t	df	Sig. (2-tailed)
Pair 1	Vita_VI - Vita_SCF	.172400	.087404	.027639	.109875	.234925	6.237	9	.000152
Pair 2	Reflex_VI - Reflex_SCF	.107000	.094757	.029965	.039215	.174785	3.571	9	.006

VI group compared to the SCF group in both veneering porcelains.
The coefficient of variation (COV) is a measure of relative variability in the same sample and expresses the ratio of the standard deviation to the mean for variance recorded between the two methods used. The COV for the Vita specimens was 6.45% (VIF) while the COV for SCF was 5.24%. For the Wieland Reflex specimens the COV was 13.3% (VIF) vs 4.61% (SCF). These findings are further reinforced by the Bland Atman Plots produced for each of the materials tested as presented in Figures 1 and 2.

Electron microscopy analysis was use to perform the fractographic analysis. Figure 3 shows a typical precrack pattern with the source of the a and 2c measurements (μm) indicated on the micrograph.

Figure 4(i) represents a typical Vickers indentation with features that are easy to discern whilst Figure 4(ii) has features which are less distinguishable. Indents that showed deflecting radial cracks or cracks that ran into voids were rejected. Similarly, radial cracks which were difficult to delineate at their tip were also rejected.

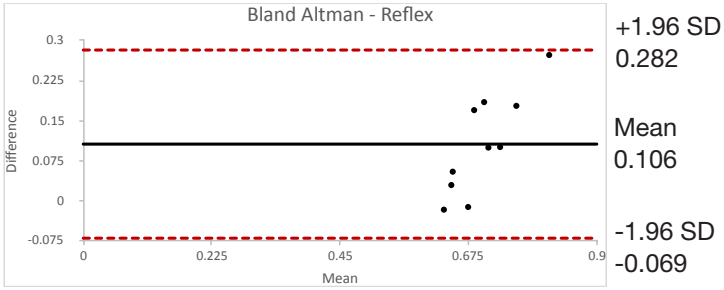

Figure 1. Plot of differences between VIF and SCF methods for Wieland Reflex Ceramic vs. the mean of the two measurements with a bias of .106 MPa $m^{1/2}$ and confidence levels set at 95%.

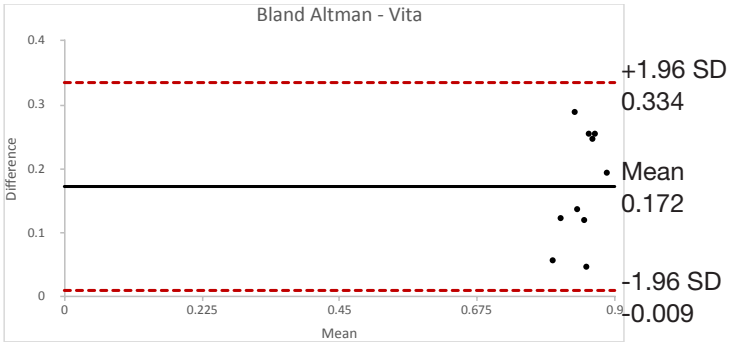

Figure 2. Plot of differences between VIF and SCF methods for Vita Ceramic vs. the mean of the two measurements with a bias of .172 MPa $m^{1/2}$ and confidence levels set at 95%.

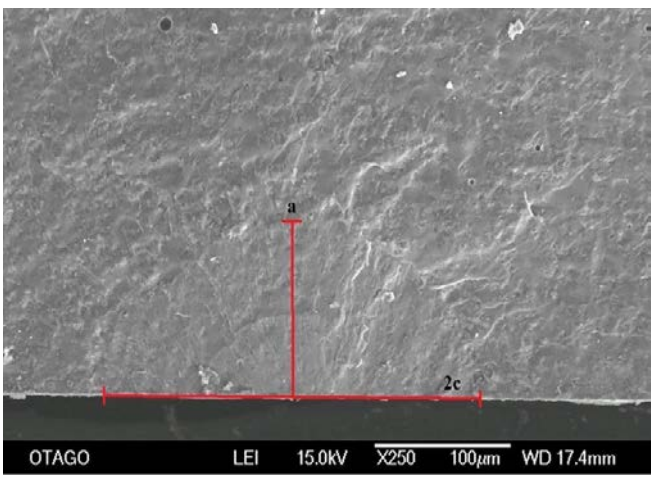

Figure 3. Scanning electron micrograph showing the precrack dimensions of a VM13 SCF specimen under 250X magnification.

Figure 4(i). Acceptable Vickers indent micrograph in Vita VM 13. Radial crack (c) and indent length (a) can distinctively be made out with no interferences (40x magnification).

Figure 4(ii). Rejected Vickers indent micrograph in Weiland REFLEX specimen. Notice the radial crack deflection due to the presence of voids shown in red (40x magnification).

Discussion

The purpose of this study was to compare the K_{IC} measurements of two veneering porcelains using two fracture toughness tests, namely the SCF and VI methods. All tests for SCF were declared valid in accordance to ASTM C1241 (2010). Statistical significance was exhibited between both methods in each veneering porcelain ($P<0.05$).

As previously indicated, the SCF technique is deemed a standardized method for calculating fracture toughness in brittle materials. ASTM C1424-10 is an American Standard Testing Method (ASTM) developed over a period time involving a myriad of experimental designs and mathematical computations to determine the most accurate method for testing, in this case, biomaterials. These standards provide the mathematical equations specifically related to the experimental design and include specimen preparation and geometry. All SCF testing in this study was done in accordance with a calibrated standardized method and thus, the values obtained for K_{IC} in both porcelain samples are considered to be valid. Quinn and Bradt (2007) found that the VI method is associated with a questionable accuracy as fracture toughness values are not coincident with those of the

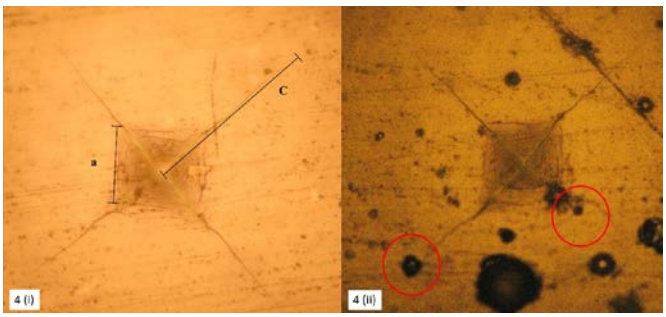

Figure 4(i). Acceptable Vickers indent micrograph in Vita VM 13. Radial crack (c) and indent length (a) can distinctively be made out with no interferences (40x magnification).

Figure 4(ii). Rejected Vickers indent micrograph in Weiland REFLEX specimen. Notice the radial crack deflection due to the presence of voids shown in red (40x magnification).

SCF method. This was substansiated in the present research and supported by the Bland Altman plots for both ceramic types. In both plots, the bias (0.106 for Reflex and 0.172 for Vita) indicates that these two methods systematically produces higher K_{IC} results, when compared to the calibrated SCF testing results. The higher coefficient of variation seen in the VIF method can possibly be related to the sheer variation and number of different equations used to calculate K_{IC} (Quinn and Bradt 2007). Over 30 equations have been developed for this method, with each giving inconsistent K_{IC} results (Evans and Charles 1976, Anstis et al. 1981, Chantikul et al. 1981, Marshall and Evans 1981, Niihara et al. 1981, Niihara 1983, Li et al. 1989, Ponton and Rawlings 1989a, Ponton and Rawlings 1989b, Ghosh et al. 1991).
A major drawback in the VI method lies in the unavoidable presence of residual stresses within the Vickers indented specimens, as well as the unstable nature and ever changing size of the cracks produced in close proximity to the indents due to environmental influence (Fischer and Marx 2002). Furthermore, the VI method is heavily criticized, due to the inaccuracy in K_{IC} values found when testing silicon nitride, which is well known as standard reference material 2100 (SRM 2100)1. The K_{IC} value from the VI test method does not match the consistent and reproducible K_{IC} values demonstrated for the material when the International Standards Organization (ISO) testing methods were used (Quinn and Bradt 2007).

Though multiple studies proved the validity of the SCF method, some researchers still prefer the "easier" VI method, purely because of the complications of the SCF method (Morrel 2006; Quinn and Bradt 2007; Cesar et al. 2017). Some of these complications are associated with the interpretation of the precracks. This can occur due to the precrack and final fracture sharing the same plane and not being tilted sufficiently to the recommended ½ degree. In such instances, the specimen may be accepted and not rejected, provided that two thirds of the precrack is visible (ASTM C1421, 2010). In other instances, variation in the appearance of precracks when viewed under SEM, can cause some complications of interpretation.

Figure 5 demonstrates more than one complication in a single image. Hand grinding or machining damage is seen in the arrows marked red at both sides of the precrack. The same image also demonstrates fine hackle lines at the crack boundary. Additionally, it shows the possibility of stable propagating crack growth, however there is

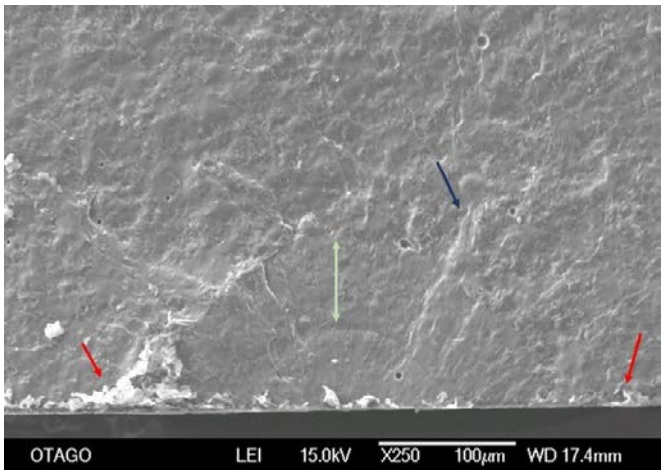

Figure 5. Micrograph demonstrating more than a single complication. Hand grinding or machining damage (red arrows, hackle lines (blue arrow) and stable crack propagation (green arrow).

inadequate evidence that this can affect the final outcome for K_{IC}. It is also difficult to truly discern stable crack extension using SEM. The same image also demonstrated hand grinding and machining damage, which can occur due to aggressive removal of the initial indent. The semi-elliptical shape is approximated and the specimen is not rejected provided that maximum Y factor is not at the surface (ASTM C1421, 2010).

The fine Hackle lines shown in Figure 3, change direction at the precrack boundary and allow for easier visualization of the precrack shape. In this study, using the backscatter function of the SEM improved depth of field of the images and thus the ability to clearly define the precrack boundaries (ASTM C1421, 2010)

Conclusion

This study measured fracture toughness of Vita VM 13 and Wieland Reflex veneering porcelains using the VIF and SCF methods. The authors found the SCF method to produce reliable results and a lower percentage variation when compared to the VI method. There appears to be a variation in K_{IC} results obtained from the two methods, with VI consistently producing higher K_{IC} values. Caution must be taken when using indentation fracture toughness methods to report absolute fracture toughness for dental porcelains and ceramics, and standardized methods should be used and adhered to.

Acknowledgements

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[1] SRM 2100 is a commercial hot-pressed silicon nitride developed by the National Institute of Standards and Technology (NIST) with a K_{IC} of 4.57 MPa $m^{1/2}$ with an uncertainty of 0.11 MPa $m^{1/2}$ at a 95% confidence level. SRM 2100 demonstrated similar and repeated K_{IC} values among all three approved ISO testing methods. However, the same was not the case when VIF was used as this method resulted in an inaccurate reporting of K_{IC}; namely K_{IC} obtained using the Anstis equation was 3.56 ±0.11 when specimens were indented at 19.6N.

References

Anstis G, Chantikul P, Lawn BR, Marshall D. 1981. A critical evaluation of indentation techniques for measuring fracture toughness I: direct crack measurements. Journal of the American Ceramic Society. 64(9):533-538.

Anusavice KJ, Shen C, Rawls HR. 2012. Phillips' Science of Dental Materials. Elsevier Health Sciences.

ASTM C1161-02c. 2008. Standard test method for flexural strength of advanced ceramics at ambient temperature West Conshohocken, PA, USA.

ASTM C1421. 2010. Standard test methods for determination of fracture toughness of advanced ceramics at ambient temperature. ASTM International, West Conshohocken, PA, USA.

Cesar P, Della Bona A, Scherre S, Tholey M, van Noort R, Vichi A, Kelly, et al. 2017. ADM guidance—Ceramics: Fracture toughness testing and method selection. Dental Materials. 33(6):575-84.

Chantikul P, Anstis G, Lawn BR, Marshall D. 1981. A critical evaluation of indentation techniques for measuring fracture toughness II: strength method. Journal of the American Ceramic Society. 64(9):539-543.

Choi JE, Waddell JN, Swain MV. 2011. Pressed ceramics onto zirconia. Part 2: Indentation fracture and influence of cooling rate on residual stresses. Dental Materials. 27(11):1111-1118.

Evans AG, Charles EA. 1976. Fracture toughness determinations by indentation. Journal of the American Ceramic society. 59(7-8):371-372.

Fischer H, Marx R. 2002. Fracture toughness of dental ceramics: Comparison of bending and indentation method. Dental Materials. 18(1):12-19.

Ghosh A, Kobayashi A, Li Z, Henager Jr C, Bradt R. 1991. Vickers microindentation toughness of a sintered SIC in the median-crack regime. Pacific Northwest Lab., Richland, WA (United States).

Guazzato M, Albakry M, Ringer SP, Swain MV. 2004. Strength, fracture toughness and microstructure of a selection of all-ceramic materials part II: zirconia-based dental ceramics. Dental Materials. 20(5):449-456.

ISO 18756. 2003. Fine ceramics. Determination of fracture toughness of monolithic ceramics at room temperature by the surface crack in flexure (scf) method. International Organization for Standardization, Geneva, Switzerland.

Li Z, Ghosh A, Kobayashi AS, Bradt RC. 1989. Indentation fracture toughness of sintered silicon carbide in the palmqvist crack regime. Journal of the American Ceramic Society. 72(6):904-911.

Marshall D, Evans A. 1981. Reply to "comment on 'elastic/plastic indentation damage in ceramics: The median/radial crack system'". Journal of the American Ceramic Society. 64(12):C-182-C-183.

Morrell R, 2006. Fracture Toughness Testing for Advanced Technical Ceramics:
Internationally Agreed Good Practice, Advanced in Applied Ceramics, 105 [2] 1–11.

Niihara K. 1983. A fracture mechanics analysis of indentation-induced palmqvist crack in ceramics. Journal of Materials Science letters. 2(5):221-223.

Niihara K, Bradt RC, Evans AG, Hasselman DP, Lange FF. 1983. Indentation Fracture Toughness of Brittle Materials for Palmqvist Cracks: Fracture Mechanics of Ceramics, Vol. 5. Plenum, NY.

Ponton C, Rawlings R. 1989a. Vickers indentation fracture toughness test part II: application and critical evaluation of standardised indentation toughness equations. Materials Science and Technology. 5(10):961-976.

Ponton CB, Rawlings RD. 1989b. Vickers indentation fracture toughness test part I: review of literature and formulation of standardised indentation toughness equations. Materials Science and Technology. 5(9):865-872.

Quinn GD, Gettings R, Kubler J. 1995. Fracture toughness of ceramics by the surface crack in flexure (SCF) method. Fracture Mechanics of Ceramics. 11:203-218.

Quinn GD. 2002. The Fracture Toughness Round Robins in VAMAS: What We Have Learned. In STP1409-EB Fracture Resistance Testing of Monolithic and Composite Brittle Materials, ed. J. Salem, G. Quinn, and M. Jenkins. West Conshohocken, PA: ASTM International.

Quinn GD, Bradt RC. 2007. On the Vickers indentation fracture toughness test. Journal of the American Ceramic Society. 90(3):673-680.

Yoshimura HN, Cesar PF, Miranda WG, Gonzaga CC, Okada CY, Goldenstein H (2005). Fracture toughness of dental porcelains evaluated by IF, SCF, and SEPB methods. Journal of the American Ceramic Society 88(6):1680-1683.

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