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Opinion piece

Indentation techniques for evaluating the fracture toughness of biomaterials and hard tissues

J.J. Kruzic^{a,*}, D.K. Kim^b, K.J. Koester^c, R.O. Ritchie^{c,d}

^a Materials Science, School of Mechanical, Industrial, and Manufacturing Engineering, Oregon State University, Corvallis, OR 97331, USA

^b Department of Materials Science and Engineering, KAIST (Korea Advanced Institute of Science and Technology), Daejeon 305-701, Republic of Korea

^c Materials Sciences Division, Lawrence Berkeley National Laboratory, Berkeley, CA 94720, USA

^d Department of Materials Science and Engineering, University of California, Berkeley, CA 94720, USA

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ABSTRACT

Indentation techniques for assessing fracture toughness are attractive due to the simplicity and expediency of experiments, and because they potentially allow the characterization of both local and bulk fracture properties. Unfortunately, rarely have such techniques been proven to give accurate fracture toughness values. This is a concern, as such techniques are seeing increasing usage in the study of biomaterials and biological hard tissues. Four available indentation techniques are considered in the present article: the Vickers indentation fracture (VIF) test, the cube corner indentation fracture (CCIF) test, the Vickers crack opening displacement (VCOD) test and the interface indentation fracture (IIF) test. Each technique is discussed in terms of its suitability for assessing the absolute and relative toughness of materials or material interfaces based on the published literature on the topic. In general, the VIF and CCIF techniques are found to be poor for quantitatively evaluating toughness of any brittle material, and the large errors involved ($\sim\pm 50\%$) make their applicability as comparative techniques limited. Indeed, indentation toughness values must differ by at least by a factor of three to conclude a significant difference in actual toughness. Additionally, new experimental results are presented on using the CCIF test to evaluate the fracture resistance of human cortical bone. Those new results indicate that inducing cracking is difficult, and that the cracks that do form are embedded in the plastic zone of the indent, invalidating the use of linear elastic fracture mechanics based techniques for evaluating the toughness associated with those cracks. The VCOD test appears to be a good quantitative method for some glasses, but initial results suggest there may be problems associated with applying this technique to other brittle materials. Finally, the IIF technique should only be considered a comparative or semi-quantitative technique for comparing material interfaces and/or the neighboring materials.

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* Corresponding author. Tel.: +1 541 737 7027; fax: +1 541 737 2600.
E-mail address: jamie.kruzic@oregonstate.edu (J.J. Kruzic).

1. Introduction

Accurately measuring the fracture toughness of brittle materials can often be challenging. Creating sharp pre-cracks can be difficult without catastrophically failing the specimen, while fracture toughness data using notched specimens can give erroneously high values (Munz et al., 1980; Ritchie et al., 1990; Fett and Munz, 2006). For those reasons, assessing fracture toughness by making direct measurements of cracks created using a sharp diamond indenter, such as Vickers, Knoop, Berkovich, or cube corner, can appear to be an attractive alternative to more traditional fracture toughness testing techniques (Lawn et al., 1980; Anstis et al., 1981; Fett, 2002; Fett et al., 2005). Such tests can be relatively quick and easy to perform, require little specialized equipment, and can allow probing of localized microstructural features. Accordingly, such techniques are finding considerable usage in studying the fracture behavior of biomaterials and hard tissues (Lopes et al., 1999; Kim et al., 2000; Marshall et al., 2001; Khor et al., 2003; Denry and Holloway, 2004; Imbeni et al., 2005; Mullins et al., 2007).

As with all fracture toughness testing techniques, the ultimate goal is to quantify the fracture toughness accurately in a way that can be universally compared with results generated using other techniques and from other studies. Unfortunately, techniques involving direct measurements from indent cracks are often unsuccessful in this regard (Li et al., 1989; Ponton and Rawlings, 1989b; Ghosh et al., 1996; Kruzic and Ritchie, 2003; Quinn and Bradt, 2007). A secondary goal may be to provide a quick semi-quantitative way to rank the toughness of different materials. In this case, indentation techniques can have some merit, but cannot be used indiscriminately. As will be discussed below, caution and good judgment are needed by the investigators, and large toughness differences are often needed to draw firm conclusions.

Accordingly, this article discusses some of the limitations of, and concerns with, using such indentation fracture techniques for studying biomaterials and biological hard tissues based on the published literature. Also presented are some new experimental results on using cube corner indentation to assess the fracture toughness of human cortical bone. Four techniques are discussed that involve making direct measurements of cracks emanating from indents. Techniques using indentation to create pre-cracks for traditional fracture toughness testing are not considered here. The techniques discussed include the Vickers indentation fracture (VIF) test, the cube corner indentation fracture (CCIF) test, the Vickers crack opening displacement (VCOD) test, and the interface indentation fracture (IIF) test.

2. Determining toughness from direct crack length measurements

2.1. Vickers indentation fracture (VIF) test

By far, the most widely used technique in the literature for assessing the fracture toughness directly from indent

cracks utilizes the Vickers indenter. First proposed in the late 1970's, this technique was developed to estimate the fracture toughness of ceramic materials by measuring the lengths of cracks emanating from Vickers indents (Lawn et al., 1980; Anstis et al., 1981). The method has subsequently received much recent attention for making measurements of fracture toughness in biomaterials (Lopes et al., 1999; Kim et al., 2000; Khor et al., 2003; Denry and Holloway, 2004). Lawn et al. (1980) modeled the elastic-plastic behavior under the indent, assuming that a median/radial crack system is created due to tensile stresses that form during unloading. They derived the expression:

$$K_c = \alpha \sqrt{\frac{E}{H}} \frac{P}{c^{\frac{3}{2}}} \quad (1)$$

where P is the applied load, E is Young's modulus, H is the hardness, and c is the length of the surface trace of the half penny crack measured from the center of the indent. α is an empirically determined "calibration" constant, taken to be 0.016 ± 0.004 based on a fit to experimental data using independent fracture toughness measurements (Anstis et al., 1981). A later model by Laugier (1985) derived a similar expression, with E/H raised to the $2/3$ rather than $1/2$ power and accordingly a different calibration constant. Published review articles on this method identify more than 30 different equations that have been presented in the literature for determining the fracture toughness from the length of the cracks observed on the sample surface (Ponton and Rawlings, 1989a; Quinn and Bradt, 2007), although most are not derived from physical models and are arrived at by curve fitting to data. Eq. (1) is generally the most cited and applied in practice and will be the focus of this discussion.

A quick review of Eq. (1), or other similar equations, reveals several causes for concern. First, an empirical calibration constant is used that has never been obtained, or justified, using physical models, and the standard deviation on the fit to obtain the calibration constant is large, $\pm 25\%$. Accordingly, at the 95% confident interval for all materials considered by Anstis et al. (1981), the toughness is only known to $\pm 50\%$ even before scatter in experimental measurements is considered. Anstis et al. (1981) only included what they considered well behaved materials in their fit, and they point out anomalous glasses or softer ceramics as possible exceptional cases when this technique will be even less accurate. Subsequent evaluations of this technique have confirmed the large errors associated with this method (e.g., Fig. 1), and have found the error in toughness to exceed $\pm 50\%$ in some cases (Li et al., 1989; Ponton and Rawlings, 1989b; Ghosh et al., 1996; Quinn and Bradt, 2007).

There are numerous potential sources that can contribute to such large errors, as there are many possible violations of the underlying assumptions of the model of Lawn et al. (1980), as well as issues such as R-curve and indentation size effects. The model assumes:

1. Two perpendicular well defined median/radial half penny cracks form below the indent during unloading due to the residual tensile stress field of the indent and,
2. The crack is, at the time of measurement, in equilibrium with that stress field, just at the stress intensity required to cause crack extension.

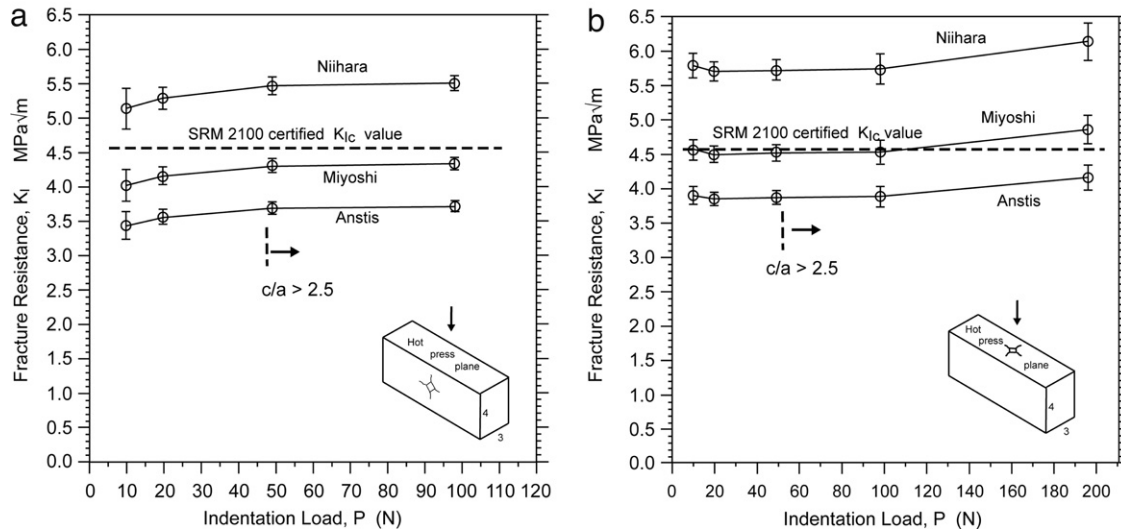


Fig. 1 – Comparisons of VIF toughness results to the actual toughness for a Si_3N_4 ceramic (SRM 2100) tested in two different orientations (see insets). Note the poor agreement between results for the three indentation equations evaluated (Anstis et al., 1981; Niihara, 1983; Miyoshi, 1985), while only in one case did the indentation toughness match the actual toughness. Similar results have been reported in many studies on many different ceramic materials, along with both increasing and decreasing apparent toughness with increasing indentation load (Li et al., 1989; Ponton and Rawlings, 1989b; Ghosh et al., 1996; Quinn and Bradt, 2007). Figure reproduced from Quinn and Bradt (2007).

In some ceramics, indentation cracking is of the Palmqvist type which is difficult to discern from the half-penny crack when only viewing the surface trace; however, the analysis of Laugier (1987) concludes that this has little effect on the fundamental fracture toughness equations, provided the cracks are in equilibrium with the stress field. Another concern is that cracks are rarely found to initiate only upon unloading, except for the case of soda-lime glass, and instead often initiate during the loading portion of the indentation process (Cook and Pharr, 1990). This has the potential to disrupt the underlying residual stress field and affect the growth of the cracks during unloading. A further concern is the formation of other cracks that disrupt the residual stress field, such as lateral cracks or cone cracks, which are also commonly observed during Vickers indentation (Cook and Pharr, 1990; Kruzic and Ritchie, 2003). This is problematic since such cracks are not readily apparent to the experimentalist in opaque materials without post indent sectioning of the sample. Cook and Pharr (1990) reported that the details of indentation cracking phenomena are extremely material dependent, explaining a large part of why an accurate equation that can be applied for all brittle materials has not been achieved. Finally, in many brittle materials, sub-critical crack growth can occur after indentation, causing the cracks to extend. This will cause an erroneously low toughness value to be calculated using Eq. (1) that may approach the sub-critical crack growth threshold depending on (1) the time between indentation and measurement and (2) the testing environment. This problem can be mostly negated by testing in an inert environment such as silicone oil, but even trace amounts of water in the testing environment can affect the results.

Another issue is found for materials that exhibit rising fracture resistance with crack extension (i.e., rising R-curve).

For those materials, only one point on the R-curve is sampled by the indentation fracture test, and that point will depend on the load used unless the R-curve plateau has already been reached. Additionally, one of the parameters in Eq. (1), H , can depend on the indentation load. H generally decreases with increasing indentation load and this effect usually saturates after some critical load (Li et al., 1989; Quinn and Quinn, 1997). In some cases this effect can be trivial and easily avoided for carefully conducted fracture tests, such as in α -SiC where the indentation size effect does not occur above ~ 3 –5 N (Li et al., 1989; Quinn and Quinn, 1997). However, in other ceramics, this effect persists up to indentation loads as high as 100 N (Quinn and Quinn, 1997). The indentation size effect can result in an apparent higher measured toughness for higher indent loads (longer cracks) even if the toughness is not actually higher, giving erroneous rising R-curve-like results even in materials with no rising R-curve. Several researchers have reported apparent increases or decreases in toughness with increasing indent load using this technique, both in materials with no rising R-curve, such as α -SiC and a ceramic glass, as well as for Si_3N_4 ceramics that should be on the plateau of the R-curve at all indent sizes tested (Li et al., 1989; Ponton and Rawlings, 1989b; Ghosh et al., 1996; Quinn and Bradt, 2007). The fact that decreasing toughness has been observed indicates that several of the above factors play a role in the apparent change in toughness with indent load. Accordingly, using varying indentation loads to measure R-curves with the VIF toughness technique should not be considered an acceptable or accurate technique.

The original data of Anstis et al. (1981), and all the subsequent data from other critical reviews (Li et al., 1989; Ponton and Rawlings, 1989b; Ghosh et al., 1996; Quinn and Bradt, 2007), agree that this method produces large errors in the measured toughness, making the VIF method inaccurate

