JOURNAL OF MATERIALS SCIENCE 37 (2002) 865–869

Indentation toughness of ceramics: A modified approach

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The indentation toughness equation proposed by Anstis *et al.* was re-examined with a particular emphasis on the definition of the hardness parameter used. It was shown that, because of the existence of the well-known indentation size effect (ISE), the apparent hardness defined as the ratio of the applied load to the projected area of the resultant indentation usually varies with the applied load and seems not to be suitable for use in indentation toughness determination. A new equation for determining indentation toughness, in which a load-independent hardness number was incorporated, was proposed.

1. Introduction

Surface cracks generated by Vickers indentation are now used extensively as model surface flaws in fracture research of brittle materials such as ceramics and glasses [1–4]. Plastic deformed zone at the center of these cracks exerts a residual crack-opening force. Such a residual stress plays an important role in the determination of the fracture toughness of brittle ceramics with indentation method. Various models for indentation residual stress have been proposed and are well summarized by Cook and Pharr [3]. Among these existing models, the Lawn-Evans-Marshall (LEM) model [5] may be the most widely cited one. The essences of the LEM model are that the well-developed indentation cracks in ceramics are half-penny in shape and the residual stress due to indentation can be regarded as being concentrated at a point located at the crack center at the elastic/plastic interface, acting as crack mouth opening point-force. Furthermore, it is assumed that the volume of the indentation plastic zone can be equated to that of an internally pressurized spherical cavity, allowing the use of Hill's solution to the expanding spherical cavity problems. On the basis of these assumptions, the radius of the cracks, c, is predicted to bear a characteristic relation to the indentation load, P

$$c = kP^{3/2} \tag{1}$$

where k is an empirically derived constant.

Equation 1, in effect, reflects an equilibrium relation between the radius of the half-penny crack and a residual crack-opening point force due to the indentation plastic zone. Based on Equation 1, Anstis *et al.* [6] proposed the following relationship for estimating or measuring fracture toughness K_C from indentation crack-length data

$$K_C = \delta\left(\frac{E}{H}\right)^{1/2} \frac{P}{c^{3/2}} \tag{2}$$

where E is Young's modulus, H is hardness and δ is a constant dependent only on the geometry of the indenter. For the standard Vickers diamond pyramid indenter, Anstis *et al.* [6] established a value, $\delta = 0.016 \pm 0.004$, by calibrating indentation parameters with fracture toughness measured by conventional fracture mechanics techniques.

Equation 2 has now been widely used for the evaluation of material toughness by indentation. However, the discrepancy between the indentation toughness determined with Equation 2 and its fracture toughness measured by other conventional methods has been frequently reported [2, 4]. The origin of this discrepancy was attributed to a variety of phenomena, including (i) the dependence of the crack geometry on the applied indentation load and the properties of the test material [7, 8] and (ii) the effect of some non-ideal indentation deformation/fracture behavior such as lateral cracking [9], subcritical growth of the indentation cracks [10], or phase-transformation due to indentation [11, 12].

In fact, another important factor that may affect the accuracy of indentation toughness determination seems to be related to the definition of hardness used in Equation 2. In the analysis of Anstis *et al.* [6], hardness was defined as the ratio of the applied indentation load to the projected area of the resultant indentation impression, i.e.,

$$H = \frac{P}{2a^2} \tag{3}$$

where a is the half-length of the diagonal of the indentation impression. However, it has been generally

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reported [13, 14] that the hardness based on such a definition, namely *apparent hardness*, is not a constant but dependent on the applied indentation load. Such a phenomenon has long been recognized as the *indentation size effect* (ISE). Clearly, the existence of the ISE makes it insufficient to quote a single hardness number when hardness is used in material characterization. Therefore, one can expect that uncertainties in the indentation toughness determined with Equation 2 may result from the use of a load-dependent hardness number.

Based on the above consideration, the effect of the apparent hardness on the indentation toughness determination with Equation 2 was examined in this paper. A modified indentation toughness equation was then proposed by incorporating a load-independent hardness number into Equation 2. The validity of this modified equation was also verified by analyzing previously published experimental data.

2. Analysis

A direct evidence for the effect of the load dependence of the apparent hardness on indentation toughness determination with Equation 2 may be provided by analyzing the variation of the experimentally measured indentation parameter $P/c^{3/2}$ with the applied indentation load. To verify the applicability of Equation 2 to the determination of indentation toughness, an extensive study was conducted by Anstis *et al.* [6]. In this study, a number of so-called "reference" materials, including glasses, a glass-ceramic, and polycrystalline ceramics, were indented with a Vickers indenter to determine the crack size, c, as a function of applied indentation load, P. It was suggested from the resultant plots of $P/c^{3/2}$ versus P that $P/c^{3/2}$ was independent of P for each material within experimental scatter. After Anstis *et al.*, the existence of a load-independent $P/c^{3/2}$-value was be widely adopted as an empirical criterion for judging whether a half-penny crack configuration is well-developed during indentation and whether Equation 2 may be suitable for calculating toughness from the indentation crack length [15–18]. However, it should be pointed out that treating $P/c^{3/2}$ as a constant may be questioned. According to Equation 2, the apparent hardness H decreases with increasing P and both K_C and E are constants for a given material. Thus it may be expected that the experimentally determined $P/c^{3/2}$ would decrease with increasing P. Fig. 1 gives the plots of the experimentally determined $P/c^{3/2}$ over the working range of indentation load P, for soda-lime glass [18] and a fine-grained alumina (grain size: 1.2 μm) [19]. A decreasing tendency in $P/c^{3/2}$ with the increasing indentation load P is evident for each material, giving a solid support for the above analysis. In fact, if the experimental results of Anstis *et al.* [6], which were given in Fig. 4 of Ref. [6], are closely examined, a decreasing tendency in the measured $P/c^{3/2}$ with increasing P can also be detected, especially for the results on Si_3N_4 (NC350), Al_2O_3 (AD999), glass (LA) and single crystal Si.

Another evidence for the effect of apparent hardness on indentation toughness determination with Equation 2 may be obtained by comparing the reported values of the constant δ appeared in Equation 2. As mentioned above, the constant δ appeared in Equation 2 has an empirical value of 0.016, which was obtained by experimental calibration with known fracture toughness values on a variety of ceramics [6]. On the other hand, a more detailed theoretical analysis by Shetty *et al.* [20] gave $\delta = 0.023$. It is well-known that the apparent hardness decreases usually with increasing indentation load and tends to a constant, sometimes referred to as the *load-independent hardness* or *true hardness*, at a relatively high load level [13, 14]. Because the apparent hardness, which is used in the experimental calibration of δ-value by Anstis *et al.* [6], is usually higher than the true hardness, which is used for the theoretical analysis by Shetty [20], it can be understood easily from Equation 2 that the experimentally calibrated δ-value is smaller than the theoretical value.

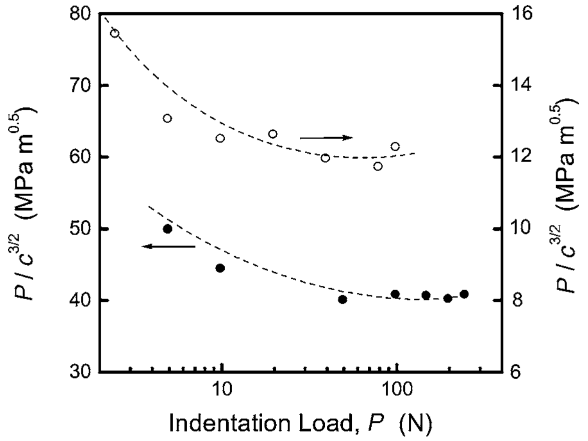

Figure 1 Plots of $P/c^{3/2}$ versus P over the working range of applied indentation load for soda-lime glass (○) [18] and a fine-grained alumina (●) [19].

Clearly, to eliminate the effect of the apparent hardness on indentation toughness determination, a load-independent hardness number should be incorporated into the indentation fracture mechanics equation. This can done by re-examining the LEM model which provides the theoretical basis for Equation 2. Following the analysis of Lawn *et al.* [5], we start with the expression for the stress-intensity factor, K_I, for a half-penny surface crack centrally loaded with a point force, F,

$$K_{\mathrm{I}} = \frac{\Omega F}{\pi c^{3/2}} \tag{4}$$

where c is crack half-length and Ω is a free-surface correction factor.

In the indentation problem, F is the residual force derived from the indentation plastic zone. Assuming the indentation plastic zone is hemi-spherical and has a radius of ρ, F is given by [20]

$$F = \int_0^{\pi/2} \pi \rho^2 \sigma_{\mathrm{r}} \sin\theta \cos\theta \, \mathrm{d}\theta = \frac{\pi \rho^2 \sigma_{\mathrm{r}}}{2} \tag{5}$$

where σ_r is the residual pressure which develops at the elastic-plastic interface as a direct result of elastic accommodation of the indentation impression volume in the hemi-spherical plastic zone. Denoting ΔV and V are the volumes of the indentation impression and the plastic zone, σ_r can be related to the elastic modulus, E, and the Poisson's ratio, υ, with the following equation

$$\sigma_r = \frac{E}{3(1-2\upsilon)}\left(\frac{\Delta V}{V}\right) \tag{6}$$

Note that $\Delta V \propto a^3$ and $V \propto \rho^3$. If we reasonably assume that the effect of Poisson's ratio can be neglected [5, 20], substituting Equations 5 and 6 into Equation 4 gives

$$K_I \propto \frac{E}{\rho}\left(\frac{a^3}{c^{3/2}}\right) \tag{7}$$

Since the parameters E, a and c can be measured easily with the conventional techniques, the key problem for using Equation 7 to estimate K_I is then the determination of the size of the plastic zone, ρ. Therefore, the relation between this radius and the elastic properties (Young's modulus, E, and Poisson's ratio, υ) and plastic properties (hardness, H_0) of the indented material has been a subject of many theoretical or experimental studies [5, 20, 21]. Here we use the empirical relation suggested by Lawn *et al.* [5] that approximates the more rigorous elastic-plastic solution proposed by Chiang *et al.* [21]:

$$\frac{\rho}{a} \propto \left(\frac{E}{H_0}\right)^{1/2} \tag{8}$$

Inserting Equation 8 into Equation 7 yields

$$K_I = \alpha(EH_0)^{0.5}\left(\frac{a^2}{c^{3/2}}\right) \tag{9}$$

where α is a constant independent of the test material.

Equation 9 predicts that it is the quantity $a^2/c^{3/2}$, rather than $P/c^{3/2}$, that would keep constant. To verify this prediction, the experimental data on soda-lime glass [18] and a fine-grained alumina [19] are now plotted in Fig. 2, where the quantity $a^2/c^{3/2}$ is shown as a function of the applied indentation load, P. Clearly, a nearly constant $a^2/c^{3/2}$ is observed for each material. Furthermore, the coefficient of variation of $a^2/c^{3/2}$ was calculated to be 0.030 for soda-lime glass and 0.023 for alumina. Compared with the coefficient of variation of $P/c^{3/2}$, 0.097 for soda-lime glass and 0.085 for alumina, the relatively smaller value of the coefficient of variation of $a^2/c^{3/2}$ seems to demonstrate that it is more reasonable to treat $a^2/c^{3/2}$, rather than $P/c^{3/2}$, as a constant.

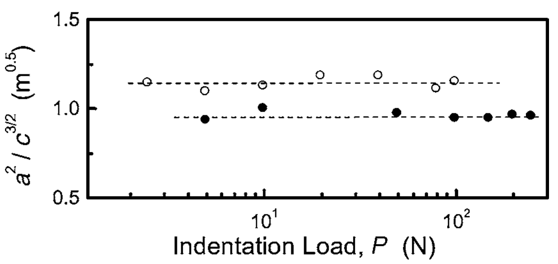

Figure 2 Plots of $a^2/c^{3/2}$ over the working range of applied indentation load P for soda-lime glass (○) [18] and a fine-grained alumina (●) [19].

The preceding analysis is similar to that conducted by Lawn *et al.* [5] for proposing their LEM model. In fact, substitution of the applied indentation load P for the indentation dimension a with Equation 3 and assuming $H \approx H_0$ would yield Equation 2, the basic equation which is now widely used in indentation toughness determination. However, it should be pointed out that such a substitution may be questioned. Equation 8 was derived directly from Hill's solution to the expanding spherical cavity problem [5, 21]. According to Hill's analysis [22], the hardness parameter, H_0, used in Equation 8 should be a material constant, which is a measure of the material resistance to plastic flow, whereas the apparent hardness, H, varies due to the existence of the ISE, as mentioned above. Thus one can expect that it is more appropriate to calculate the indentation toughness with Equation 9, rather than Equation 2.

Determining indentation toughness with Equation 9 requires prior knowledge of the load-independent hardness, H_0, of the test material. Unfortunately, up to now, a reliable method to determine the load-independent hardness has not been established yet, although extensive studies [23–26] have been conducted on this subject. Therefore, an empirical method to determine the load-independent hardness number was proposed here. Many investigators [13, 14, 27, 28] have suggested that, in the experimentally determined hardness–load curve for ceramics, a discrete transition point may exist where apparent hardness changes from being load dependent to load independent. Based on an energy balance analysis for the indentation process, Quinn and Quinn [14] proposed a new index of brittle for ceramics by combining elastic modulus and fracture toughness with the load-independent hardness measured at sufficient high indentation load level. The validity of this new index of brittleness was also discussed in detail by Quinn and Quinn [14]. Thus, there is reason to believe that the load-independent hardness, denoted hereafter as H_C, used by Quinn and Quinn is a material intrinsic parameter and may be used as an approximate estimation of the hardness H_0 appeared in Equation 9. In the following section, the applicability of this hardness number in indentation toughness determination with Equation 9 will be examined by analyzing the experimental data published previously by different authors.

3. Determination of α

The preceding section proposed a modified approach to determine the fracture toughness for brittle materials using indentation method. A key problem in using this modified approach is the determination of the parameter α appeared in Equation 9. Similar to the work of Anstis *et al.* [6], here we try to obtain an empirical value for α by experimental calibration with known fracture toughness values on a series of "reference" materials. The "reference" materials chosen for the present study are listed in Table I, alone with some information relative to indentation toughness determination.

TABLE I "Reference" materials chosen for the present study

Material[a]	E (GPa)	H_C[b](GPa)	K_{IC} ($MPa \cdot m^{0.5}$)[c]
Soda-lime glass [18]	70	4.5	0.75
Al_2O_3 (hot-pressed, grain size 1.2 μm) [19]	390	19.7	3.66
Al_2O_3 (normal sintered) [29]	272	9.3	3.8
SiC (normal sintered) [29]	410	22.2	2.2
Si_3N_4 (normal sintered) [29]	314	14.1	6.1
TZP (yttria stabilized) [30]	210	12.5	5.5
Silceram (SCF5) [31]	120	6.8	2.02
WC-5vol% Co cermet [32]	640	19.1	8.8

[a]The source references for E, H_C, and K_{IC} data are shown in parentheses.
[b]H_C was quoted approximately as the apparent hardness measured at the highest load level examined in the source references. As can be found in the source reference, such a treatment may yield a nearly load-independent hardness value defined in Section 2.
[c]The quoted K_{IC} was used in the source references as a "standard" toughness value for each material except those used in Ref. [29], which were tested with a single edge precracked beam (SEPB) method.

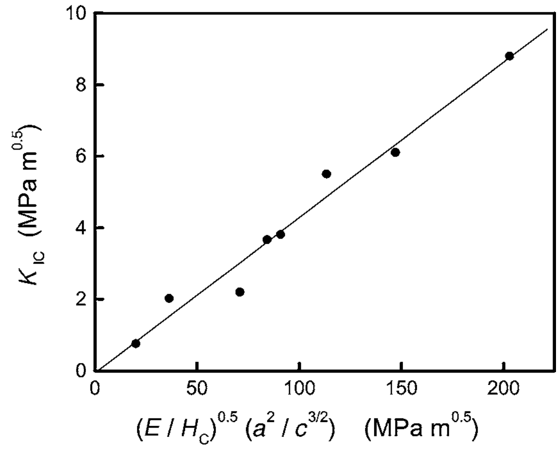

Figure 3 Plot of K_{IC} determined with conventional methods versus the quantity $(EH_C)^{0.5}(a^2/c^{3/2})$.

Careful indentation experiments on these "reference" materials have been conducted by other authors. Therefore, the following analysis was conducted directly using the indentation data reported by other authors. These data were analyzed and the relative results are summarized in Table II. Note that it was generally suggested that a indentation crack with a c/a-ratio smaller than 2.5 may not be considered to be half-penny shaped [7, 15, 16] and may deviate from the center-point loading approximation, the basis for using Equation 4 as the start equation for calculating K_I at the tip of the indentation-induced crack. The results listed in Table II were obtained only based on analyzing the original data associated with indentation cracks with a c/a-ratio larger than 2.5. For example, Ritter *et al.* tested soda-lime glass in the indentation load from 2.45 to 98.1 N. However, the indentation cracks produced at 2.45 N have an average c/a-ratio of only 2.17. Thus this data was excluded in our analysis and only the data measured in the load range from 4.9 to 98.1 N were used. Similar treatments were also conducted for some of the other materials listed in Table II.

By assuming $H_0 = H_C$, the value of the quantity $(EH_C)^{0.5}(a^2/c^{3/2})$ was calculated for each material using the data listed in Tables I and II. The calculated results are plotted in Fig. 3 as a function of K_{IC}. Fig. 3 confirms that there is a strong correlation for all investigated materials over a broad range of fracture toughness and the quantity $(EH_C)^{0.5}(a^2/c^{3/2})$. The correlation supports the analysis conducted in the preceding section. Statistical analysis of the data shown in Fig. 3 according to Equation 9 yielded

$$\alpha = 0.043 \pm 0.007 \tag{10}$$

As mentioned above, Shetty *et al.* [20] proposed a δ-value of 0.023 from a detailed theoretical analysis for deducing Equation 2. Noting the similarity between the present analysis for deducing Equation 9 and that conducted by Shetty *et al.* [20] for deducing Equation 2, one can expect that the parameter α in Equation 9 would has a theoretical value of 0.046 (i.e., 2δ). Our calibrated α-value, 0.043, is in good agreement with this theoretical result, implying that it seems to be reliable to use H_C, which was defined by Quinn and Quinn [14], approximately as the true hardness, H_0, in K_C calculation.

4. Discussion

The present modified approach to determine indentation toughness differs from the original method proposed by Anstis *et al.* [6] only in the definition of the hardness parameter used for calculation. However, it should be pointed out that such a simple modification may be important for practical applications.

In the original paper [6] where Equation 2 was proposed, Anstis *et al.* did not pay any attention to the

TABLE II Summary of the indentation data reported previously

Material[a]	Range of P examined (N)	c/a	$P/c^{3/2}$ ($MPa \cdot m^{0.5}$)	$a^2/c^{3/2}$ ($\times 1000\ m^{0.5}$)
Soda-lime glass [18]	4.9 ∼ 98.1	>2.5	12.37	1.147
Al_2O_3 (hot-pressed grain size 1.2 μm) [19]	49.1 ∼ 245.3	>3.3	39.73	0.963
Al_2O_3 (normal sintered) [29]	98 ∼ 196	>2.7	36.64	1.810
SiC (normal sintered) [29]	49 ∼ 196	>3.76	35.83	0.744
Si_3N_4 (normal sintered) [29]	196	2.545	67.07	2.212
TZP (yttria stabilized) [30]	196 ∼ 294	>2.57	60.10	2.218
Silceram (SCF5) [31]	50 ∼ 149	>3.37	19.23	1.280
WC-5vol% Co cermet [32]	294 ∼ 490	>2.83	76.23	1.837

[a]The source references for the quoted indentation data are shown in parentheses.

load-dependence of the apparent hardness, H, and simply used a constant hardness value in their experimental calibration for δ-value. This fact results in a confusion in hardness measurements in the subsequent studies concerning indentation toughness determination with Equation 2. For example, the hardness value used by Li *et al.* [7] to calculate the indentation toughness for SiC was measured with the indentations associated with microcracking, while Shi and James [33] measured the hardness at low loads where indentation-induced microcracking did not occur for their study on indentation toughness determination of glasses. Undoubtedly, such a situation would make the reported data incomparable since using different hardness values measured at different load levels would yield different values for indentation toughness. The modified approach presented in this study introduced a comparable hardness index, H_C, into indentation toughness calculation and overcome the this drawback associated with hardness measurement.

Note that H_C quoted in our modified approach is not a real *true hardness*. As mentioned in Section 2, H_C is also an apparent hardness defined with Equation 3. What we had done was only to attribute a limitation to its measurement; that is, it should be measured at relatively high load level to make it nearly load-independent. Thus, one can expect that, when measuring indentation toughness at relatively high load level, the modified approach would yield results comparable to those obtained with the original one, i.e., Equation 2. However, it is clearly appropriate to use the modified approach to determine indentation toughness in low-load regime where the ISE in the apparent hardness is usually significant [13, 14].

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Received 1 March 2000
and accepted 4 October 2001