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A Work Approach to Determine Vickers Indentation Fracture Toughness

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According to the comparison of Vickers microindentation tests and Vickers macroindentation tests on several brittle materials, it is found that the ratio of hardness (H) to elastic modulus (E) is sensitive to well-developed radial cracks, but the ratio of unloading work (W_u) to total loading work (W_t) is not. Based on this finding together with the approximate linear relationship between the ratio of H to reduced modulus (E_r) and W_u/W_t, a new approach taking W_u/W_t instead of H/E as the input parameter to determine Vickers indentation fracture toughness is proposed. For this proposed approach, all input parameters can be obtained in one single instrumented indentation test for fracture toughness, thus the test procedure can be simplified significantly. The formula of the newly proposed approach is calibrated by the macroindentation tests on several brittle materials. The validity of the new approach is investigated by comparing its estimation with the old one's.

I. Introduction

Fracture toughness is always considered as one of the most crucial mechanical properties for glasses and ceramics of which the failure mode is catastrophic. Over the past several decades, indentation fracture tests have become popular in determining fracture toughness of brittle materials at small scales.[1] In indentation fracture tests, a Vickers indenter (four-sided, having an equivalent semiconical angle of 70.3°) or a cube-corner indenter (three-sided, having an equivalent semiconical angle of 42.3°) is generally used to produce radial crack traces in the specimen surface. The cube-corner indenter is particularly used to measure fracture toughness of solids of very small volume and thin films due to its ultralow cracking thresholds.[2,3] However, in most of other cases, Vickers indenter should be the better choice. The capability of Vickers indenter to produce much larger radial cracks without causing chipping in the specimen surface makes itself excel in: (i) improving the accuracy of the measurement of radial crack lengths; (ii) reducing the influence of grain sizes of coarse materials; (iii) locating indents efficiently. One frequently used formula for determining Vickers indentation fracture toughness is given as

$$K_{IC} = \delta\left(\frac{H}{E}\right)^{-1/2}\frac{F_m}{c^{3/2}} \tag{1}$$

which was initially developed by Lawn *et al.*[4] on the assumption that radial crack traces are well-developed and half-penny cracks predominate. In Eq. (1), H and E are the hardness and elastic modulus of the test material respectively, F_m is the peak indentation load (see Fig. 1(a)), c is the length of the radial crack trace in the material surface after the indenter withdrawing (see Fig. 1(b)), and δ is an empirical constant independent of the test material. Anstis *et al.*[5] calibrated Eq. (1) by carrying out conventional indentation tests on a series of brittle materials including glasses and ceramics and obtained $\delta = 0.016 \pm 0.004$. Eq. (1) was proved still effective when shallow radial cracks other than half-penny cracks predominate by other researchers.[2,6] According to Anstis *et al.*,[5] besides the conventional indentation test, additional test is needed to measure E of the test material, which makes the test procedure complex when determining the Vickers indentation fracture toughness using Eq. (1).

Not like the conventional indentation test, the instrumented indentation test (IIT) can provide the load-depth (F-h) curve. Therefore, H and E can be calculated according to ISO 14577-1:2002[7] the total loading work W_t ($W_t = \int_0^{h_m} F dh$, see Fig. 1(a)) and the unloading work W_u ($W_u = \int_{h_p}^{h_m} F dh$, see Fig. 1(a)) can also be obtained by integrating the F-h curve. Based on the dimensional analysis and finite-element method calculations, Cheng *et al.*[8,9] found that there is an approximate linear relationship

$$\frac{H}{E_r} = k\frac{W_u}{W_t} \tag{2}$$

for the IIT without cracking in the specimen under a geometrically self-similar indenter whose equivalent semiconical angle in the range from 60° to 80°. In Eq. (2), the ratio k is independent of the test material. E_r is the reduced modulus and is calculated by

$$\frac{1}{E_r} = \frac{1-\nu_i^2}{E_i} + \frac{1-\nu^2}{E} \tag{3}$$

where E_i and ν_i are the elastic modulus and Poisson's ratio of the indenter, E and ν are those of the test material. Yang *et al.*[10] analytically found that the ratio k mainly depends on the equivalent semiconical angle of the indenter and experimentally verified Eq. (2) for the modified Berkovich indenter (three sided, having the same equivalent semiconical angle as the Vickers indenter). For the combination of the diamond Vickers indenter and most brittle materials, $E_i \gg E$, and $\nu \approx 0.25$, thus Eq. (3) reduces to $E_r = 1.07E$. Therefore, for brittle materials, Eq. (2) can be rewritten as

$$\frac{H}{E} = 1.07k\frac{W_u}{W_t} \tag{4}$$

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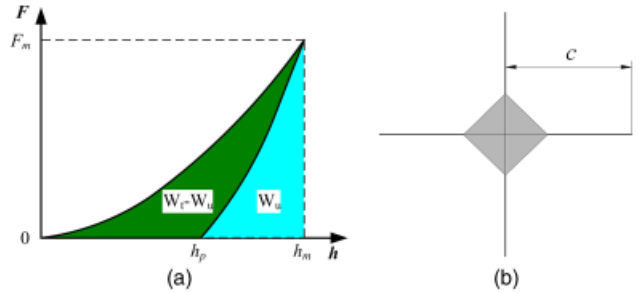

Fig. 1. Schematic diagrams of input parameters of the newly proposed approach: (a) peak load (F_m), unloading work (W_u), total loading work (W_t); (b) length of radial crack (c).

Inserting Eq. (4) into Eq. (1), we can obtain

$$K_{IC} = \lambda\left(\frac{W_u}{W_t}\right)^{-1/2}\frac{F_m}{c^{3/2}} \tag{5}$$

where $\lambda = \delta/\sqrt{1.07k}$.

It should be emphasized that there is supposed to be no cracking in the specimen when using IITs to measure H/E and W_u/W_t, respectively, for Eqs. (1) and (5). For W_u and W_t are both integral quantities, W_u/W_t may be insensitive to the occurrence of the radial cracking. If it is the case, Eq. (5) may act as the formula of a new approach to determine Vickers indentation fracture toughness of brittle materials using the IIT. And the new approach can significantly simplify the test procedure because all input parameters can be obtained in one IIT.

In this research, to estimate the influence of well-developed radial cracks on H/E and W_u/W_t respectively, micro-IITs[7] and macro-IITs[7] are carried out on several brittle materials. Micro-IITs are designed to obtain H/E and W_u/W_t in the absence of cracks. Macro-IITs are designed to obtain them in the presence of well-developed radial cracks. Furthermore, the constant λ in Eq. (5) is calibrated using the test data of macro-IITs.

II. Experimental Procedure

(1) Micro-IITs

Micro-IITs are carried out on three brittle materials, soda-lime glass, aluminosilicate glass and silicon (100), using an MTS Nano Indenter XP (MTS Nano Instruments, Oak Ridge, TN) with a diamond Vickers indenter. For each material, five peak loads, $F_m = 30, 75, 150, 300, 600$ mN, are applied. For each peak load on each material, five indentation tests are performed.

H and E are measured according to ISO 14577-1:2002. Then H/E and W_u/W_t are calculated for each material under each F_m.

(2) Macro-IITs

Macro-IITs are carried out on the same materials as those used in the micro-IITs. The patented test system[11,12] is the combination of an Instron 5848 MicroTester (Instron, Canton, MA) and an eddy current position sensor (Lianneng, Yangzhou, China) as illustrated in Fig. 2. A diamond Vickers indenter is used. For each material, five peak loads $F_m = 5, 7.1, 10, 14, 20$ N are applied. For each peak load on each material, five indentation tests are performed.

H and E are measured according to ISO 14577-1:2002. Then H/E and W_u/W_t are calculated for each material under each F_m. Radial crack lengths are measured using an Olympus BX61 optical microscope (Olympus, Shinjuku-Ku, Tokyo, Japan) after indentation.

III. Results and Discussion

Normalized H/E vs F_m and normalized W_u/W_t vs F_m over the range of micro-IITs and macro-IITs for each test material are plotted in Fig. 3. Each H/E or W_u/W_t in Fig. 3 is the mean value of five indentations for the same material under the same F_m, and is normalized by the mean value of H/E or W_u/W_t at $F_m = 30$ mN. Each error bar of H/E or W_u/W_t in Fig. 3 represents the standard deviations of the five indentations for the same material under the same F_m, and is normalized by the mean value of H/E or W_u/W_t at $F_m = 30$ mN as well. It is clearly shown in Fig. 3 that, for soda-lime glass and aluminosilicate glass, H/E keep approximately constant in micro-IITs but change significantly in maro-IITs, meanwhile W_u/W_t keep approximately constant over the whole range from micro-IITs to macro-IITs. For silicon (100), both H/E and W_u/W_t change obviously as F_m increases over the range from micro-IITs to macro-IITs, however the fluctuation of H/E is much distincter than that of W_u/W_t. It should also be noticed that the error bar of H/E is much larger than that of W_u/W_t for each material under each F_m in macro-IITs.

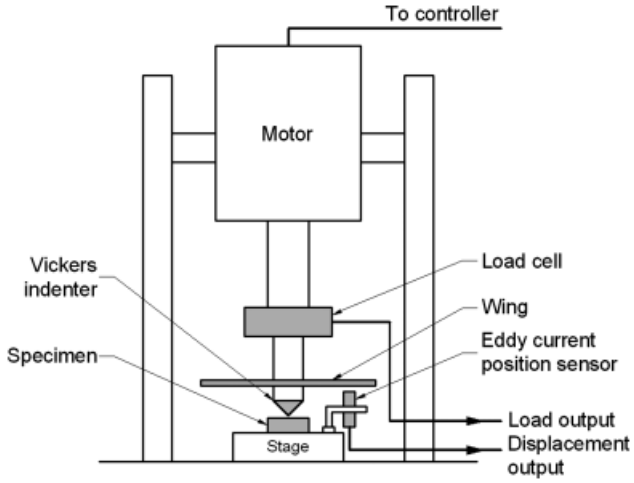

Fig. 2. Schematic diagram of the test system for macro-IITs.

Microscope observations after IITs show that, for soda-lime glass and aluminosilicate glass, no cracking or just slight radial cracking under higher F_m in specimens surfaces is observed in micro-IITs, well-developed radial crack traces under all F_m are observed in macro-IITs, and no chipping in the specimens surface is observed in both micro-IITs and macro-IITs. For silicon (100), slight radial cracking in the specimen surface is observed under most F_m in micro-IITs, well-developed radial crack traces under all F_m are observed in macro-IITs, and both are accompanied by occasional chipping in the specimen surface.

Based on Fig. 3 and the microscope observations, it can be concluded that H/E is much sensitive to well-developed radial cracks, however W_u/W_t is immune to well-developed radial cracks. Therefore, Eq. (5) is confirmed capable of determining Vickers indentation fracture toughness of brittle materials using data obtained from one single IIT. Although W_u/W_t is insensitive to well-developed radial cracks, caution should be taken in case that obvious chipping in the specimen surface emerges.

The constant number λ in Eq. (5) is independent of the material and should be calibrated by experiments. In this research, the calibration is carried out using the test data of macro-IITs on those three brittle materials. The chipping in the surface of silicon (100) is not severe, so the macro-IITs data on silicon (100) are also included in this calibration. $(W_u/W_t)^{1/2}c^{3/2}K_{IC}$ and the corresponding F_m in each macro-IIT for each material are calculated and plotted in Fig. 4. The referenced K_{IC} values of test materials are from literature (see Table I). By least-squares fitting of the test data in Fig. 4, we can obtain $\lambda = 0.0498$.

To estimate the validity of the new approach, the K_{IC} values of three test materials are recalculated using the new approach (Eq. [5] with $\lambda = 0.0498$) and using the old approach (Eq. (1) with $\delta = 0.016$), respectively, mainly based on the experimental data of macro-IITs. The K_{IC} values calculated at $F_m = 20$ N for

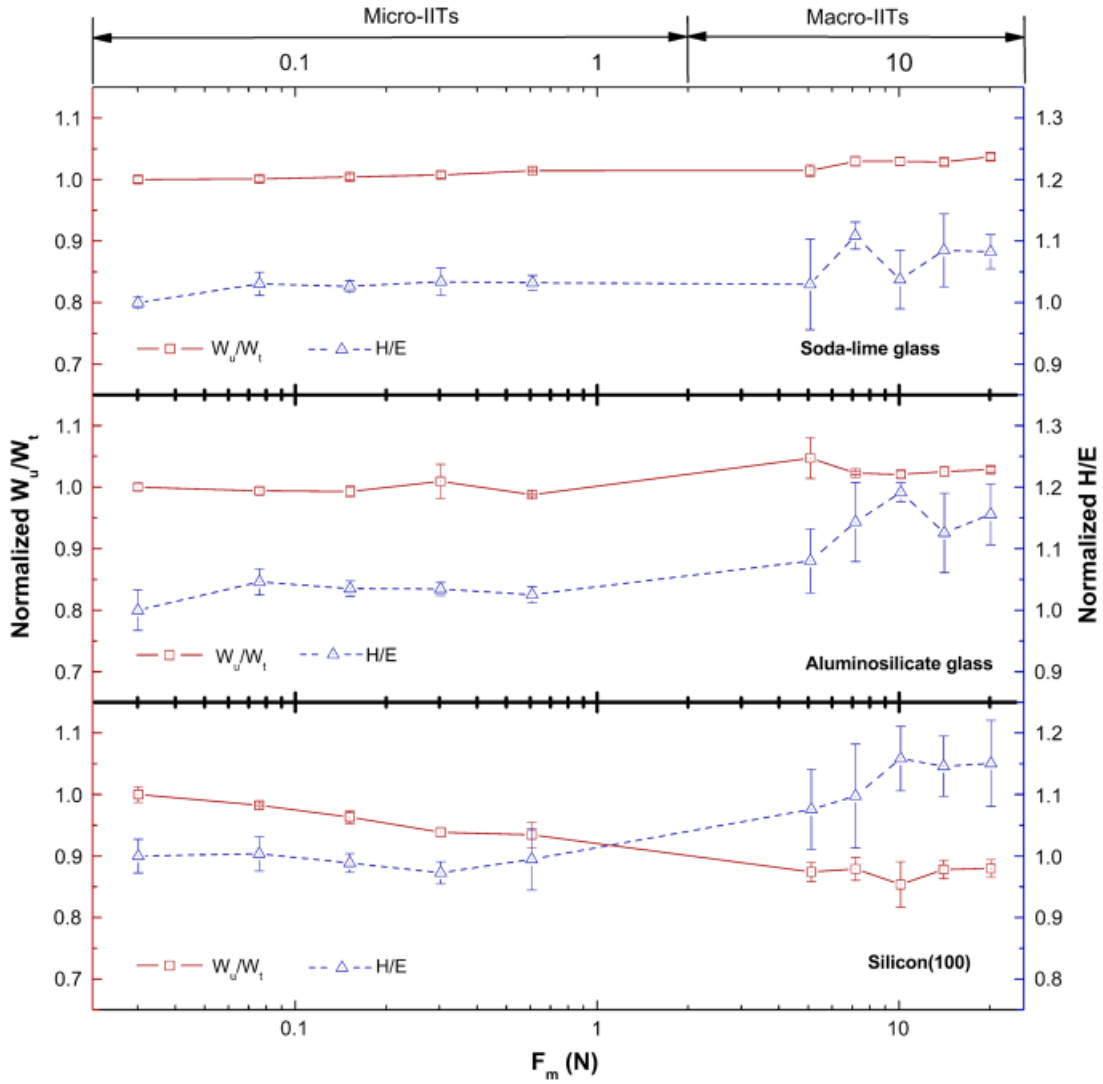

Fig. 3. Comparison of macro-IITs and micro-IITs on H/E and W_u/W_t. Each H/E or W_u/W_t in this figure is the mean value of five indentations for the same material under the same F_m, and is normalized by the mean value of H/E or W_u/W_t at $F_m = 30$ mN. Each error bar of H/E or W_u/W_t in this figure represents the standard deviation of the five indentations for the same material under the same F_m, and is normalized by the mean value of H/E or W_u/W_t at $F_m = 30$ mN as well. According to ISO 14577-1:2002 the range of micro-IITs: $F_m < 2$ N, $h > 0.2$ μm; the range of macro-IITs: $2\ \text{N} \le F_m \le 30$ kN.

three materials using the two approaches are listed in Table I, along with the referenced values from literature. Each K_{IC} value in the left two columns of Table I is the mean value of five indentations on the same material at $F_m = 20$ N. When using the old approach, H is calculated as $H = F_m/2a^2$ (a is the average half diagonal of the impression) according to Anstis *et al.*,[5] and E is calculated using the micro-IITs data according to ISO 14577-1:2002. The greatest discrepancy between K_{IC} values determined by the new and the old approaches is 19.6% which is within the coefficient of variation of the δ value in the old approach (≈25%, for $\delta = 0.016 \pm 0.004$ according to Anstis *et al.*[5]), which confirms the validity of the new approach established in this research.

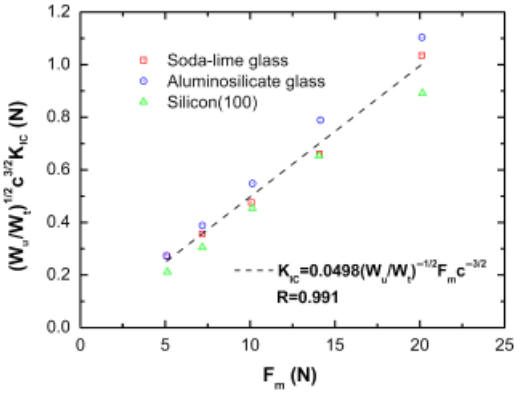

Fig. 4. Calibration of the newly proposed approach to obtain the value of λ in Eq. (5).

In this research, the number of test materials is limited and the referenced K_{IC} values used to calibrate Eq. (5) are obtained from literature. These two factors may lead to some error of λ when calibrating Eq. (5). More brittle materials with accurate K_{IC} values should be included in future research to obtain a more accurate λ value.

IV. Conclusion

In this research, according to the comparison of the Vickers micro-IITs and Vickers macro-IITs for fracture toughness on three brittle materials, we find that the presence of well-developed radial cracks without the company of obvious chipping in the specimen surface will lead to distinct error in the measurement of H/E, but not in the measurement of W_u/W_t. Based on this finding, a more convenient new approach is proposed and calibrated.

The new approach takes readily measured W_u/W_t instead of H/E as the input parameter to determine Vickers indentation

Table I. Comparison of the K_{IC} Values of Test Materials Determined by the New and the Old Approaches, Along with the Referenced Values from Literature

Material	K_{IC}—20 N (new) ($MPa \cdot \sqrt{m}$)	K_{IC}—20 N (old) ($MPa \cdot \sqrt{m}$)	K_{IC} (reference) ($MPa \cdot \sqrt{m}$)
Soda-lime glass	0.737 ± 0.053	0.616 ± 0.045	0.75†
Aluminosilicate glass	0.814 ± 0.039	0.697 ± 0.034	0.91†
Silicon (100)	0.795 ± 0.062	0.758 ± 0.050	0.7‡

†Anstis *et al.*[5] ‡Harding *et al.*[3]

fracture toughness. For this proposed approach, all input parameters can be obtained in an IIT for fracture toughness, thus the test procedure can be simplified significantly. The new approach has been proved basically effective by the comparison of K_{IC} values determined by the new and the old approaches, though broader materials collection and more accurate referenced K_{IC} values should be considered in future research to improve the accuracy of λ value.

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