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Indentation toughness of ceramics: a statistical analysis

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Abstract

Direct indentation method was employed to measure the toughnesses for soda-lime glass and a TiC particulate reinforced Al_2O_3 composite. The statistical aspect of the resultant indentation toughness was then analyzed. It was found that the indentation toughness measured at a given indentation load for a given material exhibits a large scatter and can be described with a two-parameter Weibull distribution function. Analyses based on the observations of the crack/microstructure interactions showed that the statistical variability in the measured indentation toughness may be attributed to the effect of microstructural inhomogeneity on the local crack resistance.

Keywords: C. Toughness and toughening; D. Al_2O_3; D. Glass

1. Introduction

There is considerable interest in determining the fracture toughness, K_C, of brittle ceramics and glass by measuring the extent of cracking associated with a Vickers indentation. Compared with other conventional fracture mechanics methods, the advantages of the indentation method include the small size of the test specimen, the ease of the specimen preparation, and the simplicity of the test [1]. Consequently, numerous semi-empirical equations relating material fracture toughness to the measured indentation parameters, such as the applied indentation load, final radial crack length, or indentation print size, have been derived based on experimental observations and/or theoretical considerations, see for example Refs. [1] and [2]. However, use of the Vickers indentation test to determine fracture toughness is still problematic. The discrepancy between the indentation fracture toughness of a material and its fracture toughness as measured by conventional methods, such as the single edge notched beam (SENB) method and the double-torsion (DT) method, has been reported frequently [3–6]. This discrepancy has been considered on the basis of a variety of phenomena, including: (1) the dependence of the crack geometry on the applied indentation load and the properties of the test material [7,8]; (2) the effects of some non-ideal indentation deformation/fracture behavior, such as lateral cracking [9], subcritical growth of indentation cracks [10], or phase-transformation due to indentation [11,12], and (3) unsuitable consideration of the effects of Poisson's ratio [8] and hardness [5,13].

It should be pointed out that the previous studies on the evaluation of the suitability of direct indentation method in toughness determination have thus far emphasized only the comparisons among the results obtained from different empirical or semi-empirical equations and little attention has been paid on the effect of statistical variability in the indentation toughness determined for a given material with a given equation on the reliability of these comparisons. Examining the previously reported experimental data, one can find that there always exists an inherent scatter in the indentation toughness measured at a given load, e.g. see Ref. [14]. From a viewpoint of statistics, a large scatter in a small data set would result in an inherent statistical variability in the average values obtained from different data sets. Note that different conclusions have been drawn by different authors in their studies on the comparisons between different indentation toughness equations, see for example Refs. [15–17]. There is reason to believe that the large scatter in the measured indentation toughness may be one of the important sources for these confusing conclusions.

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In this study, the indentation toughnesses of two typical brittle materials, soda-lime glass and a TiC particulate reinforced Al_2O_3 composite, were measured at different load levels and the statistical aspect of the resultant toughness data was analyzed.

2. Materials and experiments

2.1. Selection of materials

The materials selected for the present study were a commercial soda-lime glass and a 30wt.% TiC particulate reinforced Al_2O_3 composite.

The Al_2O_3 composite was fabricated in our laboratory. The basic raw materials used for preparing the composite were a commercial Al_2O_3 powder with an average grain size of 0.5 μm and TiC particles with an average size of 6.8 μm. Both powders were mixed in proper proportion by conventional ball milling with ethyl alcohol and alumina balls in a plastic pot for 4 h. After being dried, the mixed powder was uniaxially pressed at about 40 MPa and then cold isostatically pressed at about 220 MPa to form disc of about 50 mm diameter. Finally, the disc was hot-pressed at 1650 °C and 25 MPa for 30 min using a graphite die.

(1) Soda-lime glass was usually used as a reference material in the previously cited indentation fracture mechanics studies and the evolution of the half-penny crack configuration and the extension of the so-called hemi-spherical plastic zone beneath the indentation impression can be easily followed in this material [18–20]. On the other hand, our previous study [21] has shown that, for the Al_2O_3–TiC composite, the surface cracks associated with Vickers indentations made at a load higher than 49 N are also half-penny in shape. These features make it possible for us to calculate the indentation toughness, K_C, directly using the equation proposed by Anstis et al. [22]

The reasons for selecting such two materials are:

$$K_C = 0.016\left(\frac{E}{H}\right)^{1/2}\frac{F}{c^{3/2}} \quad (1)$$

where F is the indentation load, c is the half-length of the resultant indentation crack, and E and H are the Young's modulus and the hardness of the test material, respectively.

(2) Soda-lime glass is a typical brittle material with a homogeneous microstructure. On the other hand, local microstructural inhomogeneity was evident in the Al_2O_3–TiC composite due to the random distribution of TiC particles with different sizes and different shapes. A comparison between the experimental results for these two materials would be helpful for us to understand the effect of microstructure on the determination of indentation toughness.

2.2. Indentation tests

The soda-lime glass specimen used for indentation test was a plate with nominal dimensions of 50×50×3 mm. No polishing treatment was conducted for the glass specimen because its surface was mirror smooth as a result of the fabrication history. The specimen was annealed at 500 °C for 10 h to eliminate the surface residual stresses and then cleaned in an acetone bath.

The test specimens of Al_2O_3–TiC composites, with dimensions of about 4×3×20 mm, were directly cut from the hot-pressed product, mounted in bakelite, ground flat with diamond wheel and then polished carefully with successively finer diamond pastes to yield a mirrorlike surface, 4×20 mm, suitable for indentation. The polished surfaces of the test specimens are perpendicular to the hot-pressing direction. No annealing treatment was made for the composite specimens.

Vickers indentation tests were conducted in the load range from 4.9 to 98 N for soda-lime glass and from 49 to 294 N for the composite. A low-load hardness tester (Model HK-5, Wuzhong, China) was used for the indentation tests conducted in the load range from 4.9 to 19.6 N. Tests at load higher than 19.6 N were conducted with another Vickers hardness tester (model HV-120, Shandong, China). All indentation tests were performed with a constant indenter dwell time of 15 s. For the test on glass, a drop of silicon oil was placed on the prospective contact site before indentation to minimize any slow crack growth during indentation and the following measurements. Immediately after the indentation cycle had been completed, the lengths of the radial cracks were measured using an optical microscope with a magnification of 300 and an error of measurement of ±1 μm. At each indentation load level, 25 perfect indentations were made for each material.

3. Results

In general, the crack length, $2c$, used to calculate K_C with Eq. 1 was taken as the average over the two orthogonal radial directions [22]. In the present study, however, the crack lengths measured along the two orthogonal radial directions were used directly to calculate K_C, respectively. Accordingly, two K_C data were obtained for each indentation. As a result, a total of 50 K_C data were obtained at each indentation load level for each material. As will be mentioned below, such a treatment seems to be more reasonable than the traditional approach because the indentation toughness depends strongly on the local microstructural feature.

3.1. Soda-lime glass

The results of statistical analysis of the 50 K_C data obtained for soda-lime glass, including the minimum, the maximum, the arithmetic average value and its standard deviation, are summarized in Table 1. The material properties, $E=70$ GPa and $H=5.5$ GPa, used to calculate K_C with Eq. 1 were taken directly from previous studies [22,23]. As can be seen, the coefficient of variation, i.e. the ratio of the standard deviation to the average value, of the indentation toughness measured at a given applied load is usually large, typically around 10%. This result is in good agreement in magnitude with those reported by Ritter et al. [23] for the same material and reveals that the measured indentation toughness for soda-lime glass usually shows a large scatter.

Fig. 1 shows the cumulative distribution function (CDF) for the normalized K_C for soda-lime glass. In constructing this plot, the 50 K_C data measured at each load level were normalized by their average value, K_{av}, and then ordered from the lowest to the highest. The ith result in the set of 50 data, $(K_C/K_{av})_i$, is assigned a cumulative probability of failure, P_i,

$$P_i = \frac{i-0.5}{50} \tag{2}$$

The shape of the CDF shown in Fig. 1 is similar with those generally observed for the fracture strength of brittle ceramics. Thus one can assume that the statistical variability of the measured indentation toughness for soda-lime glass may be described with a Weibull distribution function, i.e.

$$P = 1 - \exp\left[-\left(\frac{K_C}{K_0}\right)^m\right] \tag{3}$$

where K_0 and m are scale parameter and Weibull modulus, respectively.

To test this assumption, a test of Pearson χ^2 was conducted and it was shown that, in each load level, the assumption that the measured K_C data follow a Weibull distribution can be accepted at a confidence level higher than 95%. Fig. 2 shows, as an example, the Weibull plots for the K_C data measured at 9.8 and 98 N, respectively. Clearly, a good linear relationship between ln K_C and ln ln $[1/(1-P)]$ was observed in each case.

Table 1
Statistical analysis results for the measured indentation toughness of soda-lime glass

Indentation load, F (N)	Indentation toughness, K_C (MPa m$^{0.5}$)				Weibull parameters	
	Minimum	Maximum	Average	Standard deviation	K_0 (MPa m$^{0.5}$)	m
4.9	0.561	0.978	0.843	0.085	0.88	11.4
9.8	0.583	0.890	0.766	0.066	0.80	13.8
19.6	0.517	0.853	0.750	0.068	0.78	12.7
49.0	0.538	0.849	0.744	0.064	0.77	14.1
98.0	0.441	0.874	0.731	0.081	0.77	12.3

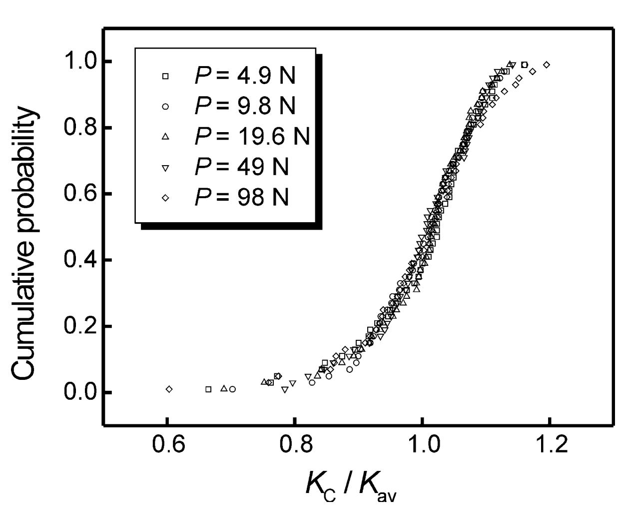

Fig. 1. Cumulative distribution function of the normalized toughness for soda-lime glass.

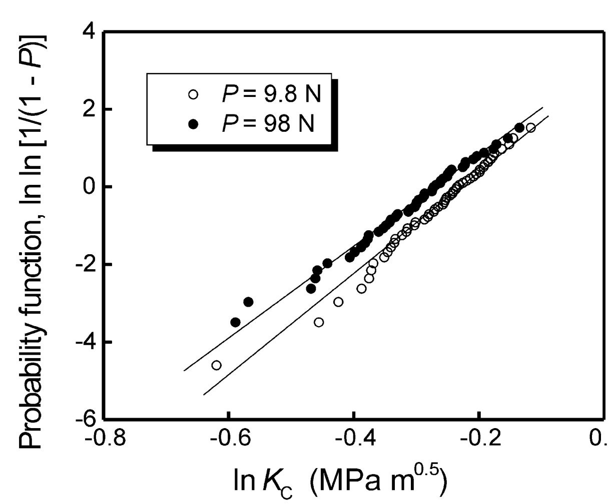

Fig. 2. Weibull plots for the indentation toughness of soda-lime glass measured at 9.8 and 98 N, respectively.

Table 2
Statistical analysis results for the measured indentation toughness of Al_2O_3–TiC composite

Indentation load, F (N)	Indentation toughness, K_C (MPa $m^{0.5}$)				Weibull parameters	
	Minimum	Maximum	Average	Standard deviation	K_0 (MPa $m^{0.5}$)	m
49	3.185	6.526	5.030	0.793	5.36	7.5
98	3.756	6.851	5.267	0.659	5.55	9.6
196	4.234	6.707	5.449	0.547	5.68	12.1
294	4.238	6.643	5.698	0.593	5.95	11.6

The Weibull parameters included in Eq. 3, K_0 and m, were estimated with the conventional least-square method [24,25] and the results are also summarized in Table 1.

4. Al_2O_3–TiC composite

The measured indentation toughness for Al_2O_3–TiC composite also shows a large scatter. Table 2 gives the results of the statistical analysis for the 50 K_C data measured at each load level for the composite and the CDFs for the K_C are illustrated in Fig. 3. The material parameter E/H (=27.40) used for the calculation of K_C for the composite was measured with a Knoop indentation method proposed by Marshall et al. [26]. The details about the measurement of E/H were reported previously [27].

Again a test of Pearson χ^2 was conducted and it was concluded that, for the Al_2O_3–TiC composite, the assumption that the K_C data measured at a given load level follow a Weibull distribution can be accepted at a confidence level higher than 95%. A least-square method was employed to estimate the Weibull parameters for each data set and the results are summarized in Table 2.

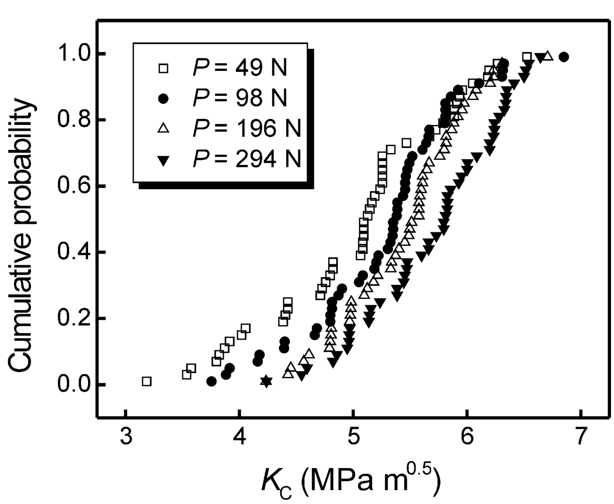

Fig. 3. Cumulative distribution function of the measured indentation toughness for the Al_2O_3–TiC composite.

As shown in Table 2, both the arithmetic average value, K_{av}, and the scale parameter, K_0, for the K_C of the composite increase with increasing indentation load. This experimental phenomenon is consistent with our previous study [27] which showed that the Al_2O_3–TiC composite examined in the present study exhibits a rising R-curve behavior, i.e. crack-growth-resistance increases as crack size increases.

5. Discussion

The main conclusion obtained in the preceding section is that, for the two materials examined, the indentation toughness measured at a given load exhibits a large scatter and can be described properly with a two-parameter Weibull distribution function. Note that soda-lime glass is a typical homogeneous material while the Al_2O_3–TiC composite can be regarded as a typical brittle material which exhibits local microstructural inhomogeneity. There is reason to believe that similar conclusion may be obtained by testing other brittle ceramics.

It is necessary to analyze the origin of the large scatter exhibiting in the measured indentation toughness. Previous studies [18,20] based on the in-situ observations of radial crack evolution during Vickers indentation have shown that the final crack configuration is achieved as the indenter is removed from the specimen surface and the driving force for crack growth is provided by a residual stress field which results from the mismatch between the plastic zone beneath the indentation and the surrounding elastic matrix. According to the theory of indentation fracture mechanics, this driving force can be expressed as:

$$K_r = \frac{\chi_r F}{c^{3/2}} \tag{4}$$

where χ_r is a dimensionless constant dependent on the materials properties and the indenter geometry.

Eq. 4 shows that, during the unloading half-cycle of an indentation event, the driving force decreases with increasing crack size. When the crack size, c, increases to a characteristic value, say c_0, the driving force, K_r, would be equal to the fracture toughness, K_C, of the test materials and the crack system reaches its final configuration, i.e. the mechanical equilibrium state. This is the theoretical basis for the determination of fracture toughness with direct indentation method [22].

Note that the above analysis is based on an assumption that the considered material can be regarded to be isotropic and homogeneous. Such an assumption may be valid for most brittle ceramics and glasses when being examined in the macroscopic scale. When examined in the microscopic scale, however, this assumption may be questioned. It is well known that, in a real solid, there always are different types of microstructural defects, such as pores, grain boundaries, inclusions, surface damages, etc. Undoubtedly, an extra energy dissipation would be expected when an indentation crack propagates to intersect with these microstructural defects, thereby resulting in the change in the total fracture energy or the toughness. This seems to be a possible explanation for the scatter observed in the measured indentation toughness.

A sound support for the above analysis may be provided by observing the crack path in the surface of the Al_2O_3–TiC composite specimen. Fig. 4 shows the propagation paths of different cracks in the surface of the composite specimen. In the Al_2O_3 matrix (the dark phase), the crack path is somewhat straight and crack deflection and crack branch may be detected occasionally. These observations can be attributed to the fact that the Al_2O_3 grain size in the composite is small, about 1–3 μm, and has little effect on the crack behavior [28]. When the crack propagates to intersect with the TiC particles (light phases), however, the features of crack/microstructure interactions are rather evidently different. Several typical features were observed: (1) the crack propagates directly through the TiC particle with little deflection; (2) the crack propagates directly through the TiC particle with significant deflection; and (3) the crack propagates along the Al_2O_3–TiC interface. Thus, one can expect that the different cracks shown in Fig. 4 would encounter different resistances due to the difference in details of the interactions between crack and microstructure.

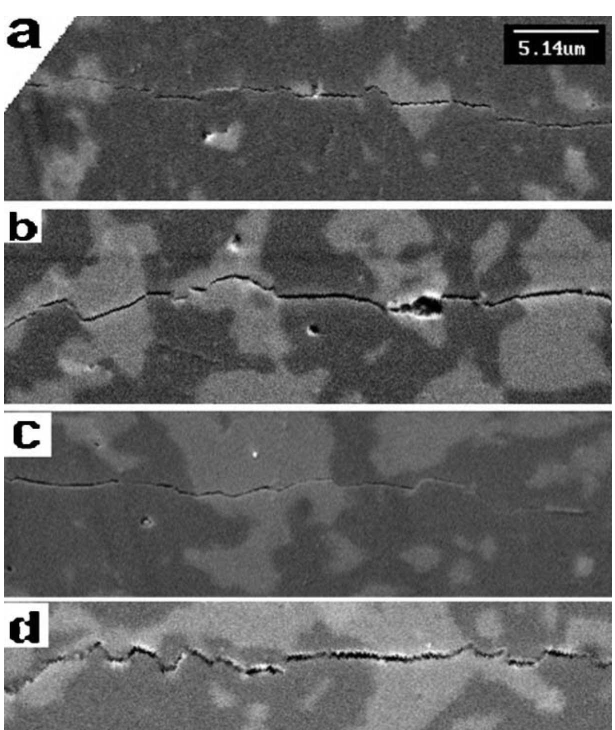

Fig. 4. Typical morphologies of the propagation paths of different indentation cracks in the surface of Al_2O_3–TiC composite.

It is of interest to note that the Weibull parameter, m, for the measured indentation toughness of Al_2O_3–TiC composite increases with the increasing indentation load and then tends to be invariable when the indentation load is high enough (see Table 2). Such an experimental phenomenon can be understood easily. When the indentation load is low, the resultant indentation crack is small. Thus the probability for different cracks to encounter different microstructural features is large. Consequently, the scatter in the crack size, hence the indentation toughness would be large, resulting in a small m-value. As the indentation load increases, a large crack would be obtained and the probability for different cracks to encounter different microstructural features would decrease. As a result, the scatter in the measured indentation toughness would decrease and m increase and finally reach a plateau level.

Following the above analysis, one can conclude that the fracture toughness measured with the direct indentation method is different to those measured with the conventional fracture mechanics techniques such as SENB or DT. In the conventional techniques, a macroscopic artificial crack, usually as large as several millimeters in length, is employed and the resultant toughness should be the material property in a macroscopic scale. On the other hand, due to the smallness of the indentation-induced crack, the indentation toughness is only a microscopic property, i.e. the local crack resistance, and depends strongly on the details of the microstructure the crack encountered.

Now we turn to analyze the experimental results for soda-lime glass. As mentioned above, soda-lime glass is a typical brittle material with a homogeneous microstructure. Several good quality micrographs showing the propagation paths of indentation cracks in glass surface can be found in the literature. The propagation paths of indentation cracks in the glass surface have been proved to be straight in general. However, local microstructural inhomogeneity may also be detected in the soda-lime glass. At least, due to the intrinsic brittleness, small-scale contact damage is always inevitable in the glass surface. Therefore, as a measure of the local crack resistance, the scatter in the measured indentation toughness seems not to be surprising. Note that the Weibull modulus, m, for the measured indentation toughness for soda-lime glass is independent of the applied indentation load and has a relatively larger value when compared with those for the Al_2O_3–TiC composite. This can be attributed to the fact that the glass is much closer to the ideally isotropic, homogeneous material than the composite.

6. Summary and remarks

The preceding analysis and discussion reveal that the measured indentation toughness for brittle ceramics and glasses may exhibit a large scatter. Such a large scatter may be attributed to the effect of microstructural inhomogeneity on the local crack resistance. In fact, indentation toughness is only a microscopic property which is rather different with the fracture toughness measured with conventional fracture mechanics techniques.

It was found that a two-parameter Weibull distribution function may be employed to describe the statistical variability of the indentation toughness measured at a given applied load. Weibull statistics has been widely used in the evaluation of the strength characteristics for brittle materials [24,25]. At present, it is impossible to test if there is a correlation between the Weibull modulus for strength analysis and that for indentation toughness analysis, although their physical origins are similar with each other.

Extensive studies [25,29] have recommended that, as a compromise between obtaining narrow confidence limits and economic considerations, a minimum of 30 repeated tests are required for an accurate estimation of the Weibull modulus for strength characterization. Similarly, it is strongly suggested that, when employing the direct indentation method to determine K_C, at least 30 independent measurements should be made at randomly selected sites in the specimen surface, in order to get a complete characterization of the resultant indentation toughness.

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