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Refined measurements of indentation fracture resistance of alumina using powerful optical microscopy

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Abstract

A round-robin of the indentation fracture (IF) method using two alumina ceramics was performed in 12 laboratories to confirm the significantly improved reproducibility of indentation fracture resistance K_{IFR}, using powerful optical microscopy. Powerful optical microscopy with both an objective lens of 40 × or 50 × and a traveling stage was employed to reduce the error in reading crack length. Indentations at 98 N for the two samples had moderate between-laboratory standard deviations of 0.3 and 0.2 MPa $m^{1/2}$ for K_{IFR} of 4.3 and 3.6 MPa $m^{1/2}$, respectively, which indicates the effectiveness of this measurement technique to improve the reliability of the IF method. The deviations of the grand average K_{IFR} reported by the laboratories from those re-measured by the authors using the returned samples were only ca. 0.4 MPa $m^{1/2}$, which was attributed to the slight misreading of the crack length by the participant laboratories. Thus, the reliability of the IF method seems reasonable by this advanced approach because our recent round-robins, together with this study, have confirmed that the precision for the three major structural ceramics, SiC, Si_3N_4 and alumina, could meet the necessary condition of reproducibility.

Keywords: C. Toughness and toughening; D. Al_2O_3; Indentation fracture technique

1. Introduction

The worldwide market of many small ceramic products and components such as bearing balls and cutting tools has been growing rapidly [1]. Evaluation of the fracture toughness of such parts is necessary for both assessment of the grade of such products and their quality control [2,3]. However, conventional standards for the toughness test are difficult to apply because the sizes of these products are smaller than those of the test specimens required for these standard methods. For example, the length of the test piece must be larger than 18 mm for single edge-precracked beam (SEPB) [4,5] and surface crack in flexure (SCF) methods [6]. One alternative technique to measure the fracture toughness of small ceramic parts is the indentation fracture (IF) method. This method is particularly useful when the sizes of available specimens are limited because only a small flat portion of a smooth surface is required. Therefore, this method has been widely used for determining the apparent fracture toughness of ceramics since it was proposed by Lawn et al. [7]. Both the ISO 26602 international standard and ASTM F 2094 American standard have adopted the IF method as a classification tool for the grading of silicon nitride bearing balls [2,3]. ISO 14627 specifies the experimental procedure and the term "indentation fracture resistance, K_{IFR}" is defined for the apparent fracture toughness because there have been rigorous arguments that the value measured using the IF method does not represent the real fracture toughness [8–10].

However, the IF method has been generally regarded as an inferior technique because the reproducibility between laboratories from round-robin tests conducted about two decades ago (e.g., VAMAS [11–14]) was very poor, although this technique is still frequently used in industry. Misreading of the crack lengths has been conjectured as a plausible reason for the large scattering; however, there have been few systematic studies reported to confirm the origin of the variation, and this has been one of the major obstacles to the standardization of the IF method. Our preliminary study on the possible errors of the IF method has clarified that the subjectivity of the operator during crack length measurement is the major cause of the wide scatter of K_{IFR} measured by different operators in our own laboratory [15]. The poor consistency between laboratories for

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the Si_3N_4 sample with some porosity was explained by misreading of the crack length in our previous international round-robin test [16]. For the solid-phase sintered SiC sample, the same difficulty in detecting the real crack tips was also found to be responsible for the large variation of K_{IFR} in our recent domestic round-robin with 10 laboratories [17]. To resolve such uncertainty, powerful optical microscopy with both an objective lens of 40 × or 50 × and a traveling stage was selected for the crack-length measurement because it is apparent that high resolution of the crack-tip image enables more precise identification of its position. Excellent reproducibility was demonstrated in round-robins using both SiC and Si_3N_4 samples and our improved test method [17,18].

The applicability of the new method to other major ceramics should be verified to expand the scope of the ISO 14627 international standard for the IF method, which is applicable only to bearing grade Si_3N_4 ceramics. Alumina is one of the most widely used structural ceramics whose very poor coefficient of variance (COV) in K_{IFR} of up to 20% has been also reported [14]. In this study, the validity of our refined technique was examined for two types of alumina ceramics with different purities through round-robin testing by 12 laboratories in Japan. The participants consisted of six universities, four companies and two national laboratories. All of the participants observed the indentations with an objective lens of 40 × or 50 × . The crack length measurements were calculated according to the shift of the microscope stage because the cracks extended over the range of the microscope. After measurements were completed by each laboratory, the test specimens were returned to the authors and the indentations were re-measured with a powerful optical microscope to determine the origin of the K_{IFR} scattering among the laboratories. Slow environmentally-assisted crack growth of the two alumina samples was hardly detected in our preliminary study. Therefore, the re-measured crack lengths were compared directly with those reported in the round-robin test. The effectiveness of the developed approach with respect to precision was compared with the precision of those reported for both SiC and Si_3N_4 samples in our previous round-robins [17,18], and this is discussed in conjunction with the low visibility of crack tips due to weak contrast.

2. Experimental procedure

2.1. Materials

Two types of alumina ceramics from commercial sources, sample A with a purity of 99.6 mass% (Hi-Cera HA, Mitsui Mining & Smelting Co., LTD., Tokyo, Japan) and sample B with a purity of 96.9 mass% (SSA-96, Nikkato Co., LTD., Tokyo, Japan), were employed as common samples for the round-robin indentation tests. The characteristic properties of the two samples, such as bulk density, relative density, grain size and Young's modulus, are summarized in Table 1. The bulk density was measured with the Archimedes technique and the relative density was calculated using the theoretical density of 3.987 g/cm^3. Young's modulus was obtained by the ultrasonic pulse echo method. The range of grain size was determined using micrographs of the polished and thermally etched surfaces. The relative density of sample A was slightly higher than that of sample B, which resulted in a slightly higher Young's modulus for A. The grains sizes of the two samples were almost the same. The fracture toughness K_{Ipb} of samples A and B obtained using the SEPB method [5] was 3.6 ± 0.2 and 3.3 ± 0.1 MPa m$^{1/2}$, respectively.

Table 1
Properties of the two alumina ceramics used in this study.

Material code	Purity (mass%)	Bulk density (g/cm^3)	Relative density (%)	Grain size (μm)	Young's modulus (GPa)
A	99.6	3.88	97.4	1–20	370
B	96.9	3.83	96.3	1–20	354

Rectangular specimens were machined from both sintered A and B samples. The sizes of the A and B specimens were $21 \times 12 \times 3$ mm^3 and $34 \times 5 \times 3$ mm^3, respectively. The larger surface was ground with a #400 diamond wheel and then polished using 0.5 μm diamond slurry on a tin plate to obtain a mirror finish for indentation tests. Optical microscopy revealed many small black dots on the mirror finished surfaces of both samples (Fig. 1), which were intrinsic pores and the result of grain fragmentation during the polishing process. All the samples were prepared by the authors and then delivered to 12 laboratories in Japan for indentation and crack length measurements.

Post-indentation slow crack growth (SCG) was evaluated by the authors preliminary to the round-robin. Crack lengths of eight and five indentations at 98 N were measured for the A and B samples, respectively. The time interval between unloading and measurement was varied from 2 to 44640 min (1 month). The time dependence of the mean crack length after unloading is presented in Fig. 2. No significant SCG was observed for the A sample and that of the B sample was negligible at only ca. 5 μm after 1 month.

2.2. Test procedure

Many invalid indentations with unacceptable crack morphologies were produced at 196 N in our preliminary study. Therefore, to improve the success rate, the indentation force was reduced to 98 N. The indentation contact time was 15 s. More than eight Vickers impressions were made at each laboratory with a hardness tester. Only indentations where four primary cracks emanated straightforward from each corner were accepted. Indentations with badly split cracks or with gross chipping or spalling were rejected, in addition to those with asymmetrical cracks.

Both a traveling stage and a powerful microscope were employed by the 12 laboratories to measure the size of indentations. The magnification of the objective lens for laboratory nos. 1–3 was 50 × and that for the remaining was 40 × , except for laboratory no. 10, which used a digital microscope with the objective lens integrated into the system, so that only the total magnification was available. However,