Available online at www.sciencedirect.com

ScienceDirect

Ceramics International 40 (2014) 2777–2783

CERAMICS INTERNATIONAL

www.elsevier.com/locate/ceramint

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

Hiroyuki Miyazaki*, Yu-ichi Yoshizawa

National Institute of Advanced Industrial Science and Technology (AIST), Anagahora 2266-98, Shimo-shidami, Moriyama-ku, Nagoya 463-8560, Japan

Received 3 July 2013; received in revised form 8 October 2013; accepted 9 October 2013
Available online 18 October 2013

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

*Corresponding author. Tel.: +81 52 736 7486; fax: +81 52 736 7405.
E-mail address: h-miyazaki@aist.go.jp (H. Miyazaki).

0272-8842/$ - see front matter © 2013 Elsevier Ltd and Techna Group S.r.l. All rights reserved.
http://dx.doi.org/10.1016/j.ceramint.2013.10.039

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,

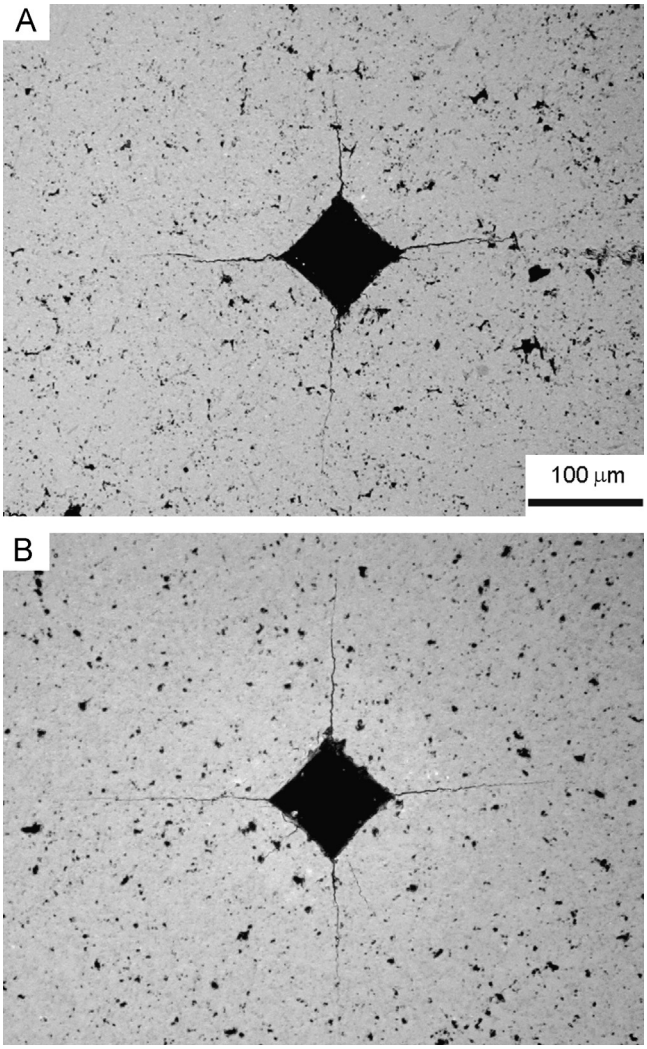

Fig. 1. Optical micrograph of an indentation made at 98 N.

such recent optics had sufficient resolution. Four laboratories out of 11 observed impressions directly with an eyepiece of 10×, so that the total magnification was 500× (laboratory nos. 1–3) or 400× (laboratory no. 4). The other seven laboratories attached both a CCD camera and a monitor to the microscope instead of an eyepiece, and their total magnification ranged from 900× to 1320×, excluding laboratory no. 12, where the magnification was only 325×. The total magnification used at laboratory no. 10 was 1000×.

The lengths of the impression diagonals, $2a$, and surface cracks, $2c$, were measured within 10 min after indentation by the participants, excluding laboratory no. 3, to minimize the effect of SCG. The time interval between unloading and the observation took more than 30 min for laboratory no. 3 because the indenter and the powerful optics were located in separate buildings.

K_{IFR} was determined from the as-indented crack lengths using Niihara's equation for the median crack system [19]:

$$K_{IFR} = 0.0309(E/H)^{2/5} P c^{-3/2}, \quad (1)$$

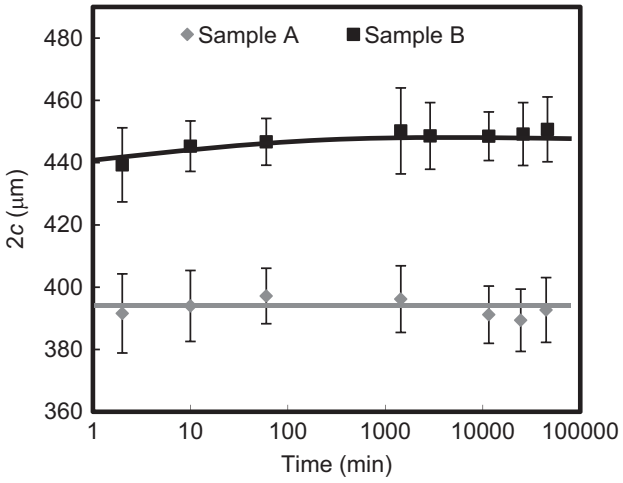

Fig. 2. Mean crack length, $2c$, as a function of log time for the two alumina samples tested in air (indent force $P=98$ N). The error bars represent one standard deviation.

where E and H are Young's modulus and the Vickers hardness, respectively, P is the indentation force, and c is the half-length of the as-indented surface crack length. In this study, Young's modulus listed in Table 1 was used. K_{IFR} was calculated for each indentation using the hardness value obtained for each impression. The calculated K_{IFR} together with the raw data was collected by the test organizer.

All samples indented by each laboratory were returned to the author's laboratory (AIST) for re-evaluation of the indentation sizes. It was deemed that a good resolution could be obtained with a measuring microscope, by which the crack tips were detected at a high magnification of 500× and the spacing between the tips was measured preciously using the traveling stage with a readout resolution of 1 μm. Thus, a measuring microscope with an objective lens of 50× (total magnification: 500×) was selected for the re-measurements. In some cases, the numbers of indentations measured by each laboratory were slightly different from those checked by AIST, due to the subjective judgment of acceptable crack morphology.

3. Results and discussion

3.1. Sample A

The crack lengths, $2c$, measured with 40× or 50× objective lenses are plotted in Fig. 3 (closed circles). The variation of the crack lengths was in the range of 340–397 μm and the grand average of $2c$ from all laboratories was 374 ± 18 μm. The crack lengths checked at AIST with the measuring microscope are also shown (open triangles) in Fig. 3. The $2c$ values were almost constant at 394 ± 15 μm among the laboratories, which indicates that the actual $2c$ values were not significantly influenced by the use of different indenters, as reported by the authors previously [20]. The difference of $2c$ between each laboratory value and the re-measurement by AIST was only ca. 20 μm for most of the participants. Only three laboratories read the crack length as

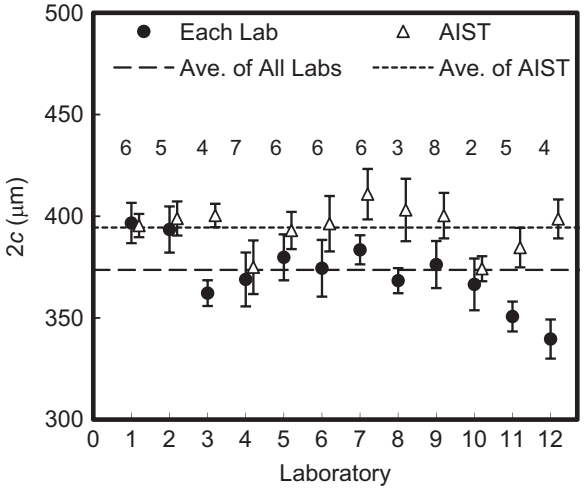

Fig. 3. Crack length, 2*c*, of the alumina A sample measured with the objective lens of 40 × or 50 × . The numbers of specimens measured by each laboratory are shown. The error bars represent one standard deviation.

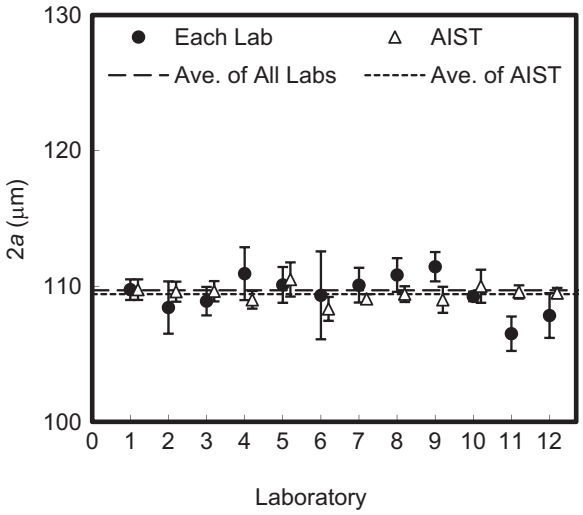

Fig. 4. Diagonal size, 2*a*, of the alumina A samples indented at 98 N. The error bars represent one standard deviation.

30–40 μm shorter. A significant misreading of more than 40 μm was reported by laboratory no. 12, which could be accounted for by insufficient magnification on the TV monitor. Fig. 4 shows the diagonal sizes, 2*a*, reported by each laboratory (closed circles) and checked at AIST (open triangles). The 2*a* values from the participants were almost constant and did not differ significantly from those re-measured at AIST.

Fig. 5 shows K_{IFR} calculated using the 2*a* and 2*c* values. Most of the values of K_{IFR} (closed circles) from the participants, excluding laboratory no. 12, were in the range of 3.9–4.6 MPa $m^{1/2}$ and the scattering was moderate. The data points of K_{IFR} re-measured at AIST were slightly shifted to the lower side. The grand averages of the fracture resistance reported by all laboratories and those from AIST are shown as dashed and dotted lines, respectively. These results indicate that the deviation of the participants' values from those of AIST was only 0.35 MPa $m^{1/2}$. Therefore, the K_{IFR} values from each laboratory are not as inaccurate as those reported in the previous round-robins [11–14].

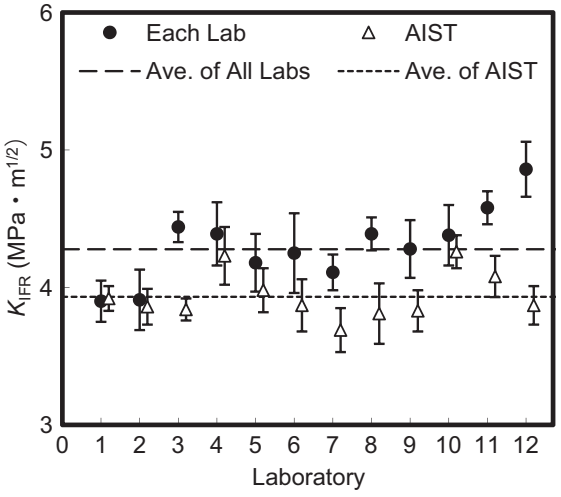

Fig. 5. Results of the round-robin on indentation fracture resistance for the alumina A samples. The error bars represent one standard deviation.

3.2. Sample B

The raw data for the crack lengths are presented in Fig. 6 (closed circles). A relatively small variation in the crack lengths from 406 to 439 μm was obtained by most laboratories, except laboratory nos. 8 and 10. The difference in the grand average for 2*c* from all the participants and that re-measured at AIST was only 35 μm, which indicates that the misreading of 2*c* by the participants was not so significant when compared with those shown in the literature [11–14]. One possible reason for the misreading of 2*c* is that the magnification of the objective lens used by most laboratories was 40 × and slightly lower than that used at AIST. The binocular microscope used by the authors at AIST enabled clearer observation of crack tips than those from the monocular microscopes used by the participants. In addition, the operator at AIST was well-trained and had a greater experience in finding the crack tip than the operators at each laboratory, which would also contribute to the difference in the 2*c* measurements. The diagonal sizes reported by each laboratory are shown in Fig. 7 (closed circles) together with those from AIST (open triangles). The 2*a* values reported by the participants were almost identical to those measured by AIST, excluding laboratory no. 10.

Fig. 8 shows that K_{IFR} (closed circles) obtained by each laboratory, except laboratory nos. 8 and 10, is in relatively good agreement and in the range 3.4–3.7 MPa $m^{1/2}$. The average of all the reported K_{IFR} was 3.63 ± 0.24 MPa $m^{1/2}$ (dashed line), which differed slightly from the values re-measured by AIST (3.21 ± 0.13 MPa $m^{1/2}$, dotted line). The reproducibility of K_{IFR} was notably improved in comparison with those of previous studies, where the COV was ca. 20% [11–14], and became acceptable with the new measuring

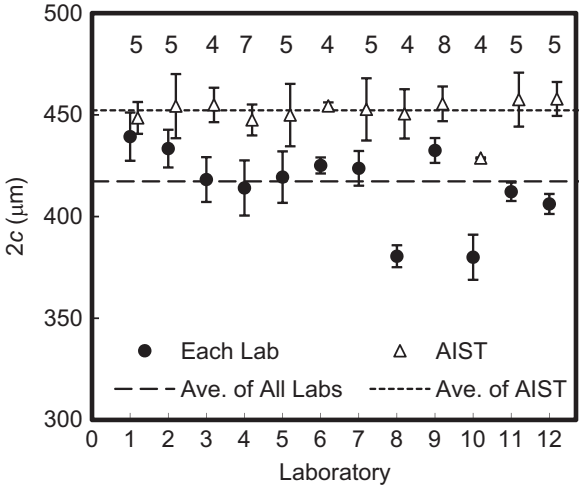

Fig. 6. Crack length, 2*c*, of the alumina B samples measured with the objective lens of 40 × or 50 × . The numbers of specimens measured by each laboratory are shown. The error bars represent one standard deviation.

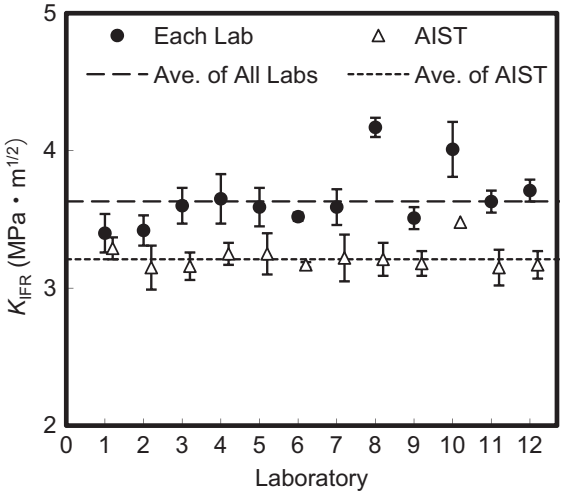

Fig. 8. Results of the round-robin on indentation fracture resistance for the alumina B samples. The error bars represent one standard deviation.

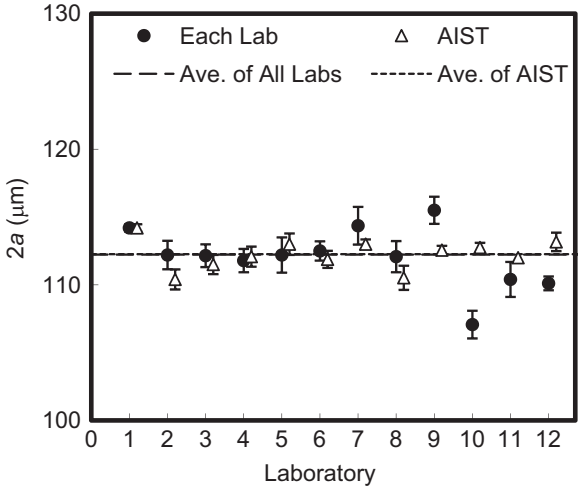

Fig. 7. Diagonal size, 2*a*, of the alumina B samples indented at 98 N. The error bars represent one standard deviation. In the figure, the dotted line is obscured by the dashed line.

technique, although there still remains room for improvement of the accuracy. The deviation of the averages received from each laboratory from that re-measured at AIST was attributable to slight misreading of the crack length by the participants.

The results were analyzed numerically in accordance with the Japanese Industrial Standard Z8402-2 [21] to evaluate the accuracy of the measurement methods, and the results are shown in Table 2. The repeatability characterizes the variance of the results within each laboratory, i.e., the variance of the results obtained by the same operator with the same equipment in a short period of time. The reproducibility describes the dispersion of the results among the laboratories. It was revealed that the COV of the data reported by all laboratories for the two samples was reasonable at ca. 7%, although the COVs were still slightly higher than the 4–6% obtained at AIST. The difference between the average K_{IFR} from all laboratories and that re-measured K_{IFR} at AIST was 0.35 MPa $m^{1/2}$ for sample A and 0.42 MPa $m^{1/2}$ for sample B. The accuracy may thus be insufficient from an academic viewpoint and so further advancement in the test methods is necessary. In contrast, the precision seems to be sufficient for practical industrial applications because it is comparable to that measured for a hot isostatically pressed (HIPed) Si_3N_4 sample by the surface crack flexure (SCF) method [6], with a COV of 9%, which was demonstrated by an international round-robin test [22]. Thus, the reliability of K_{IFR} for alumina could be improved in comparison to those reported by previous round-robins through the adoption of both an objective lens of 40 × or higher and a traveling stage. The reason for the refined measurements with a high magnification objective lens is due to its high resolving power, as explained in our previous paper [17].

3.3. Comparison of improved precision among Si_3N_4, SiC and alumina

The COV of K_{IFR} for Si_3N_4, SiC and alumina samples in our round-robin tests is compared in Fig. 9. COVs for the two alumina samples were 3% worse than those for Si_3N_4 and SiC, even when measured using the new technique. The inferior reproducibility for the alumina samples is attributable to the irregular crack lengths reported by a few laboratories, such as laboratory no. 12 for sample A and laboratory nos. 8 and 10 for sample B. The origin of such irregular data points from a limited number of laboratories can be explained by the visibility of the crack.

In the case of black ceramics such as SiC and Si_3N_4, the illumination used in the microscope is reflected from the mirror finished surface and the incident ray does not diffuse below the sample surface. Therefore, no reflected light from the crack goes into the objective lens and the crack contrast is clear because the diffuse reflection at the crack tips is negligible. In contrast, the incident ray diffuses below the surface of white ceramics such as alumina and zirconia due to their

Table 2
Accuracy of the IF method based upon the round-robin results according to JIIS Z 8402-2 for two alumina samples indented at 98 N [21]. Results re-measured by AIST are also included for comparison.

Sample	Observer	Labs	Total Indents	Average (MPa $m^{1/2}$)	Repeatability (within-lab)		Reproducibility (between labs)	
					Std. Dev. (MPa $m^{1/2}$)	COV[a] %	Std. Dev. (MPa $m^{1/2}$)	COV[a] %
A	Each lab	12	62	4.28	0.20	4.7	0.32	7.4
	AIST	12	73	3.93	0.16	4.1	0.23	5.8
B	Each lab	12	61	3.63	0.12	3.4	0.24	6.6
	AIST	12	58	3.21	0.12	3.6	0.13	3.9

[a]Coefficient of variance.

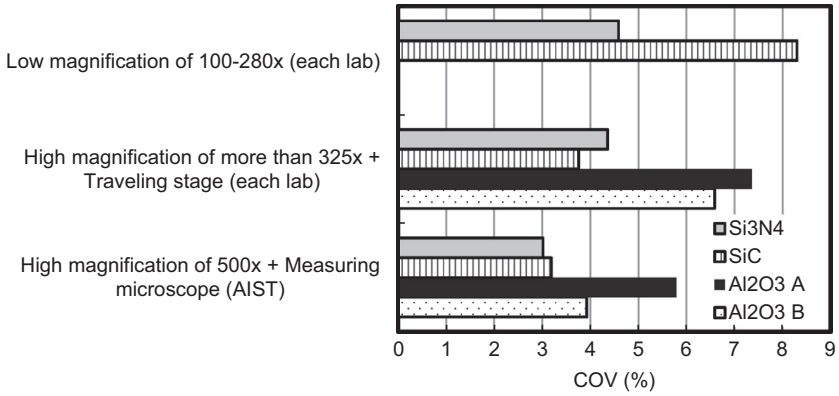

Fig. 9. COV of K_{IFR} for Si_3N_4, SiC and alumina samples measured using various apparatuses. The data for Si_3N_4 and SiC were obtained from the literature [17,18].

translucency, which results in diffuse reflection from the crack. Therefore, the contrast in a crack image of alumina is decreased due to diffuse reflections. Thus, it can be supposed that the visibility of the crack tip is inferior for alumina samples due to the inferior contrast of the crack. Accordingly, the refinement of two point resolution using a powerful objective lens is not sufficient to diminish misreading of the crack length for alumina; therefore, enhancement of the contrast at the crack is also necessary.

This approach to obtain an accurate K_{IFR} must be valid for most alumina ceramics because the general purity of alumina ceramics ranges from 95% to 99.9%, which is almost covered by the two samples used in this study. Our previous two round-robins together with this study have clarified that the reproducibility of K_{IFR} for three major engineering ceramics, Si_3N_4, SiC and alumina, has become equivalent to those of standard toughness tests, such as the SCF method, when using the refined method. Accordingly, it is reasonable to conclude that the IF method has reached a satisfactory level.

4. Conclusion

A domestic round-robin test on the K_{IFR} of two types of alumina ceramics with high and low purity was conducted in 12 laboratories. Both a traveling stage and an objective lens of $40\times$ or $50\times$ were employed to reduce misreading of the crack length from indentations made at 98 N. The same indentations on the returned samples were evaluated at AIST for comparison with the values reported by each participant. The following results were obtained:

(1) Most of the participants gave almost the same K_{IFR} value and only a few laboratories reported irregular values. The grand averages of K_{IFR} for the two samples reported by the participants were 4.28 ± 0.32 and 3.63 ± 0.24 MPa $m^{1/2}$, which demonstrates a significant improvement in the reproducibility of the test results compared with those of the previous round-robin tests conducted approximately two decades ago.
(2) The grand averages of K_{IFR} for both samples from all participants were ca. 0.4 MPa $m^{1/2}$ higher than those of the K_{IFR} re-measured at AIST, due to the slight misreading of the $2c$ by each laboratory.
(3) The reliability of K_{IFR} for the alumina samples using the proposed technique was not as good as that for the SiC and Si_3N_4 samples in our recent round-robins. The lack of improvement in precise crack-tip detection of the alumina samples could be accounted for by the weak contrast of the crack image due to the diffuse scattering of incident rays below the surface of the test piece.

(4) The refined IF method could be regarded as reliable because it can overcome the large dispersion of data, as reported for our recent round-robins.

Acknowledgments

The authors express sincere thanks to all the participants involved in this round-robin test. The authors also acknowledge the workshop on innovation for reliability in bulk ceramics of the Ceramic Society of Japan and its organizer, Assistant Professor K. Yasuda of the Tokyo Institute of Technology.

References

[1] K. Komeya, Material development and wear applications of Si_3N_4 ceramics, Ceram. Trans. 133 (2002) 3–16.
[2] Fine Ceramics (Advanced Ceramics, Advanced Technical Ceramics)—Silicon Nitride Materials for Rolling Bearing Balls, International Organization for Standards, ISO 26602, 2009.
[3] Standard Specification for Silicon Nitride Bearing Balls, ASTM F 2094/M2094M, 2008.
[4] T. Nose, T. Fujii, Evaluation of fracture toughness for ceramic materials by a single-edge-precracked-beam method, J. Am. Ceram. Soc. 71 (1988) 328–333.
[5] Fine Ceramics (Advanced Ceramics, Advanced Technical Ceramics)—Test Method for Fracture Toughness of Monolithic Ceramics at Room Temperature by Single Edge Precracked Beam (SEPB) Method, International Organization for Standards, ISO 15732, 2003.
[6] Fine Ceramics (Advanced Ceramics, Advanced Technical Ceramics)—Determination of Fracture Toughness of Monolithic Ceramics at Room Temperature by the Surface Crack in Flexure (SCF) Method, International Organization for Standards, ISO 18756, Geneva, 2003.
[7] B.R. Lawn, A.G. Evans, B. Marshall, Elastic/plastic indentation damage in ceramics: the median/radial crack system, J. Am. Ceram. Soc. 63 (1980) 574–581.
[8] Fine Ceramics (Advanced Ceramics, Advanced Technical Ceramics)—Test Method for Fracture Resistance of Silicon Nitride Materials for Rolling Bearing Balls at Room Temperature by Indentation Fracture (IF) Method, International Organization for Standards, ISO 14627, 2012.
[9] G.D. Quinn, Fracture toughness of ceramics by the Vickers indentation crack length method: a critical review, Mechanical Properties and Performance of Engineering Ceramics II: Ceramic Engineering and Science Proceedings, 27, John Wiley & Sons, Hoboken, NJ, USA, 2006.
[10] G.D. Quinn, R.C. Bradt, On the Vickers indentation fracture toughness test, J. Am. Ceram. Soc. 90 (2007) 673–680.
[11] D.M. Butterfield, D.J. Clinton, R. Morell, The VAMAS Hardness Round-Robin on Ceramic Materials, VAMAS Report #3, National Physical Laboratory, Teddington, Middlesex, United Kingdom, 1989.
[12] H. Awaji, T. Yamada, H. Okuda, Result of the fracture toughness test round robin on ceramics—VAMAS Project, J. Ceram. Soc. Jpn. 99 (1991) 417–422.
[13] H. Awaji, J. Kon, H. Okuda, The VAMAS Fracture Toughness Test Round-Robin on Ceramics, VAMAS Report #9, Japan Fine Ceramic Center, Nagoya, Japan, 1990.
[14] Report of Preliminary Investigation for Standardization of Fine Ceramics, Japanese Fine Ceramics Association, Japan, 1998.
[15] H. Miyazaki, H. Hyuga, Y. Yoshizawa, K. Hirao, T. Ohji, Study of factors affecting the length of the surface crack in silicon nitrides introduced by Vickers indentation, Ceram. Eng. Sci. Proc. 28 (2007) 391–398.
[16] H. Miyazaki, Y. Yoshizawa, K. Hirao, T. Ohji, Indentation fracture resistance test round robin on silicon nitride ceramics, Ceram. Int. 36 (2010) 899–907.
[17] H. Miyazaki, Y. Yoshizawa, K. Yasuda, Round robin on indentation fracture resistance of silicon carbide ceramics by using a powerful optical microscope, Ceram. Int. 39 (2013) 611–617.
[18] H. Miyazaki, Y. Yoshizawa, K. Yasuda, Improved accuracy of the measurements of indentation fracture resistance for silicon nitride ceramics by the powerful optical microscopy, Ceram. Int. 39 (2013) 9499–9504.
[19] K. Niihara, R. Morena, D.P.H. Hasselman, Evaluation of K_{Ic} of brittle solids by the indentation method with low crack-to-indent ratios, J. Mater. Sci. Lett. 1 (1982) 13–16.
[20] H. Miyazaki, H. Hyuga, Y. Yoshizawa, K. Hirao, T. Ohji, Measurement of indentation fracture toughness of silicon nitride ceramics, Key Eng. Mater. 352 (2007) 45–48.
[21] Accuracy (Trueness and Precision) of Measurement Methods and Results—Part 2: Basic Method for the Determination of Repeatability and Reproducibility of a Standard Measurement Method, Japanese Industrial Standard, JIS Z 8402-2, 1999.
[22] G.D. Quinn, R.J. Gettings, J.J. Kübler, Fractography and the surface crack in flexure (SCF) method for evaluating fracture toughness of ceramics, Ceram. Trans. 64 (1996) 107–143.