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Ceramics International 39 (2013) 9499–9504

CERAMICS INTERNATIONAL

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Improved accuracy of the measurements of indentation fracture resistance for silicon nitride ceramics by the powerful optical microscopy

Hiroyuki Miyazaki[a,*], Yu-ichi Yoshizawa[a], Kouichi Yasuda[b]

[a]*National Institute of Advanced Industrial Science and Technology (AIST), Anagahora 2266-98, Shimo-shidami, Moriyama-ku, Nagoya 463-8560, Japan*
[b]*Department of Metallurgy and Ceramics, Graduate School of Science and Engineering, Tokyo Institute of Technology, 2-12-1-S7-14 Ookayama, Meguro-ku, Tokyo 152-8552, Japan*

Received 21 March 2013; accepted 16 May 2013
Available online 23 May 2013

Abstract

Thirteen laboratories participated a round robin to investigate the reproducibility of indentation fracture resistance, K_{IFR}, of silicon nitride ceramics. When the usual optical microscope with an object lens of ~10 × was employed for the crack-length measurements, K_{IFR} varied from 7.7 to 8.8 MPa m$^{1/2}$, whereas those re-measured by authors using the measuring microscopy with an objective lens of 50 × exhibited a constant value of 7.5 ± 0.2 MPa m$^{1/2}$. The inaccurate K_{IFR} of each laboratory was attributed mainly to the misreading of the crack length ~30 μm shorter. By contrast, powerful microscopy with both an object lens of 40 × and a traveling stage for the crack-length reading enabled each lab to measure more accurate K_{IFR} of 7.8 ± 0.3 MPa m$^{1/2}$. The usefulness of the new measuring technique for the higher reliability of K_{IFR} was also confirmed for the Si_3N_4 ceramics just like those reported for SiC ceramics previously.

Keywords: C. Toughness and toughening; D. Si_3N_4; Indentation fracture technique

1. Introduction

Determination of fracture toughness for many small ceramic products and components such as bearing balls and cutting tools is necessary for both assessments of the grade of the products and quality control [1,2]. However, conventional toughness evaluation methods are difficult to apply since the sizes of these products are smaller than the test specimens needed in these standard methods. For example, the length of the test piece must be larger than 18 mm for single edge-precracked beam (SEPB) [3,4] and surface flaw in flexure (SCF) methods [5]. One of the alternative techniques 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 since it needs only a small portion of flat surface. Then it has been widely used for determining apparent fracture toughness of ceramics since it has been proposed by Lawn and his co-workers [6].

However, the trials to standardize the IF test have been unsuccessful since there have been rigorous arguments that the value measured by the IF method does not represent the real fracture toughness [7,8]. Some experts of toughness measurements of ceramics insist that the term “indentation fracture resistance, K_{IFR}” should be used when the IF method is applied [7]. Thus, both the international standards ISO 14627 and ISO 26602 and the American standard ASTM F 2094 for the evaluation of silicon nitride bearing balls adopt the term “indentation fracture resistance, K_{IFR}” for the apparent fracture toughness measured by the IF method [1,2,9].

The second serious issue to be solved for the standardization of the IF method is the poor reproducibility between the laboratories, which was clarified by round-robin tests about 2 decades ago (e.g. VAMAS etc. [10–13]). However, the origin of the wide scatter among the laboratories has not been explored systematically so far. Our preliminary study on the factors affecting the precision of the IF method indicated that

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

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http://dx.doi.org/10.1016/j.ceramint.2013.05.068

the error in measuring crack length was the main cause of the wide scatter of K_{IFR} between the different operators in our laboratory [14]. The international round-robin test with six laboratories using Si_3N_4 samples revealed that the misreading of crack length by each laboratory resulted in the wide variation in K_{IFR} when the sample with some porosity was measured [15]. The poor between-laboratory consistency was predominant for SiC in our domestic round robin with 10 laboratories [16], which was also attributed to the difficulty in detecting the real crack tips with the poor microscope such as the one equipped with the hardness tester. In order to reduce such human error, we have developed a new measuring technique for crack length by employing both the powerful microscopy with the object lens of 40 × or 50 × and the traveling stage. It was demonstrated that the excellent reproducibility could be obtained by this method in the second round robin using the same SiC samples [16].

In this study, mirror-finished samples prepared from commercial silicon nitrides were delivered to 13 laboratories in Japan to confirm the validity of this new technique for other engineering ceramics. The participants consisted of six universities, five companies and two national laboratories. The round-robin test was conducted twice with the same indented samples but by the different measuring methods. At the first round robin, almost all labs used an optical microscope with a low magnification of 100–280 × . After the measurements by each labs, the test specimens were returned to the authors and their indentations were re-measured with a powerful optics in order to find out which was the origin of the scatter of K_{IFR}, that is, the difference of the real size of indentations itself or the systematic biases of measurement due to different operators. It has been reported that the environmentally-assisted slow crack growth was negligible for Si_3N_4 [14,17,18] and K_{IFR} hardly changed after unloading [14]. Then, the re-measured crack lengths were compared directly with those reported values in this round-robin test.

In the second round-robin test, all the participants observed the indentations at high magnifications of 400 × or higher by using an objective lens of 40 × . The distances of crack tips were determined from the shift of the stage of the microscope since the cracks extended over the range of the microscope. The effectiveness of the developed technique on the correct measurements was compared with those reported on SiC in our previous round robin and discussed with conjunction of the width of crack very near to the crack tips.

2. Experimental procedure

2.1. Materials

Silicon nitride ceramics from commercial source (SN-1, Japan Fine Ceramics Center, Nagoya, Japan) were employed as the common sample for the round robin. The bulk density of the sample was 3.20 g/cm^3 and Young's modulus obtained by the ultrasonic pulse echo method was 284 GPa. The microstructure consisted of needle-like grains, which was described elsewhere [15]. Rectangular specimens with dimensions of 4 mm × 3 mm × 38 mm were machined from the sintered samples. The larger 4 mm × 38 mm surface was polished to a mirror finish for indentations.

2.2. Test procedure

More than eight Vickers impressions were made at each laboratory with the hardness tester. The indentation load was 196 N and the indentation contact time was 15 s. The lengths of the impression diagonals, $2a$, and surface cracks, $2c$, were measured immediately after the indentation. Only indentations whose four primary cracks emanated straightforward from each corner were accepted. Indentations with badly split cracks or with gross chipping were rejected as well as those asymmetrical ones.

Eleven labs out of 13 employed an objective lens of 10 × , 13 × (lab no. 3) or 20 × (lab no. 13) to measure the size of indentations at the first round robin and two labs (lab nos. 1 and 5) used much higher magnification of 50 × for the object lens. In order to investigate the effect of the low magnification on the measurements, the data from the former labs were analyzed in this paper. The microscopes of nine labs were furnished with their testers. Six labs out of nine observed impressions directly with an eyepiece of 10 × , so that the total magnification was 100 × , 130 × (lab no. 3) or 200 × (lab no. 13). Lab nos. 4, 9 and 12 attached both a CCD camera and a monitor to the microscope instead of an eyepiece, which resulted in a little bit higher magnification of 250 × or 280 × . The measurements with a measuring microscope or a metallurgical microscope were performed by lab nos. 6 and 11 and their total magnifications were 100 × .

The indentation fracture resistance, K_{IFR}, was determined from the as-indented crack lengths by Niihara's equation for the median crack system as follows [19]:

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

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

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

K_{IFR} reported by each lab exhibited large scattering and was different from those re-measured value by the authors, implying that the detection of exact crack tip was difficult with the poor microscope. It has been proved that higher magnification of the object lens was useful to find the real crack tips in our previous round robin with the SiC sample [16]. Then, the second round-robin test using a microscope equipped with both a powerful objective lens and a traveling stage was conducted in order to improve the accuracy of the measurements. The indented samples were sent to 12 laboratories again and the indentations made in the first round-robin test were re-measured using both an objective lens of 40 × and a traveling stage. Half of the labs observed the impressions directly at a total magnification of 400 × with a eyepiece of 10 × . A CCD camera and a monitor were employed by the rest of labs to obtain total magnifications of 600 × or higher.

3. Results and discussion

3.1. First round robin using a low magnification

The crack lengths, $2c$, measured at a low total magnification of 100–280 × are plotted in Fig. 1 (closed square). The variation of the crack lengths was in the range of 358–389 μm and the grand average of $2c$ was 373 ± 11 μm. The crack lengths re-measured by authors with the measuring microscope are shown as open triangles in Fig. 1, as well. Almost constant and longer $2c$ values of 393 ± 8 μm were observed among the laboratories, indicating that real $2c$ themselves were hardly influenced by the different indenters as reported by authors previously [20]. The comparison of $2c$ between each lab's value and our re-measured one revealed that all the participants read the crack length little bit shorter than the test organizer when they employed the low magnification and that their misreading of $2c$ ranged from 4 to 30 μm. It was suggested that finding exact crack tips at the low magnification was difficult as compared to the powerful microscopy with higher magnifications. Fig. 2 shows the diagonal sizes, $2a$, reported by each lab and re-measured by the authors (closed square and open triangle). Participants' $2a$ were almost constant and did not differ significantly from those re-measured by authors.

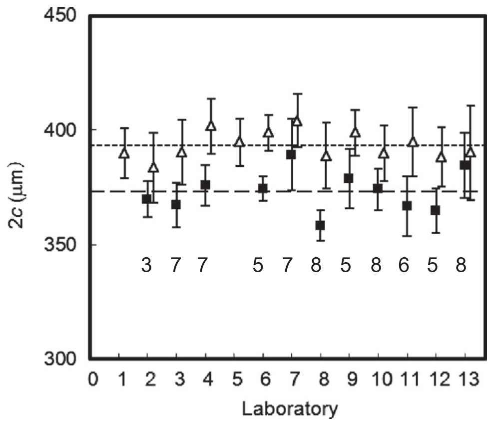

Fig. 1. Crack length, $2c$, of the Si_3N_4 samples measured at the first round robin. Closed squares represent each laboratory's average observed at a lower magnification from 100 × to 280 × , while open triangles denote those re-measured by authors with a measuring microscope at a total magnification of 500 × . Number of specimens measured by each laboratory and one standard deviation (error bars) are also shown. The dashed line represents the average of all the reported data, whereas the dotted line is the average of all the re-measured ones by authors.

Indentation fracture resistance, K_{IFR}, was calculated with these $2a$ and $2c$ and are shown in Fig. 3. Relatively large scatter of K_{IFR} (7.7–8.8 MPa $m^{1/2}$) was observed among the laboratories (closed square), while re-measured K_{IFR} by authors exhibited constant values of 7.5 ± 0.2 MPa $m^{1/2}$ (open triangle). The ground average of the fracture resistance reported by each lab and the authors are shown as dashed and dotted lines, respectively, which indicated that the average of participants' values was 0.7 MPa $m^{1/2}$ higher than that of the authors. It is obvious that each lab's K_{IFR} was not precise and that the major origin of such uncertainty was the error in reading crack length.

3.2. Second round robin using both an objective lens of 40 × and a traveling stage

In order to improve the accuracy of the measurements of crack length, the objective lens with a high magnification of 40 × and the traveling stage were employed at the second round robin. Silicon nitride specimens were sent back again to each laboratory and the same indentations made at the first round robin were measured by each lab. The raw data of crack lengths is presented in Fig. 4 (closed circle). Relatively small variation in the crack lengths from 372 to 393 μm was obtained when measured by this technique, which was improved as compared with that of the first round robin at the low magnification. The difference of $2c$ between each lab's value and our re-measured one was reduced by almost half of those of the first round robin, indicating that the misreading of $2c$ by the participants decreased. One of the possible reasons of the remaining error in $2c$ is that the magnification of objective lens used by each lab was slightly lower than that of ours. The

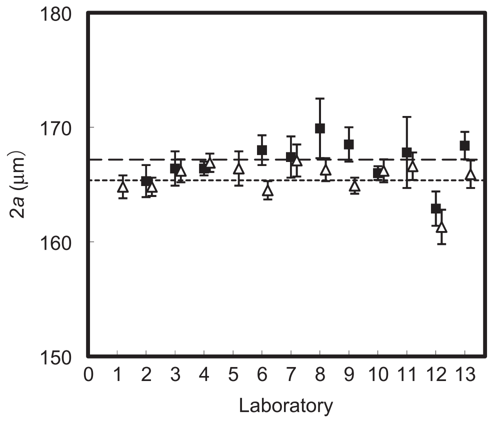

Fig. 2. Diagonal size, $2a$, of the Si_3N_4 samples measured at the first round robin. Closed squares represent each laboratory's average observed at a lower magnification from 100 × to 280 × , while open triangles denote those re-measured by authors with a measuring microscope at a total magnification of 500 × . One standard deviation (error bars) is shown. The dashed line represents the average of all the reported data, whereas the dotted line is the average of all the re-measured ones by authors.

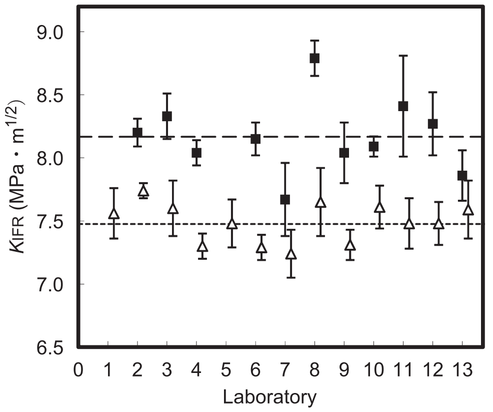

Fig. 3. Results of the first round robin on indentation fracture resistance of the Si_3N_4 samples. Closed squares represent each laboratory's average observed at a lower magnification of 100–280 ×, while open squares denote those re-measured by authors with a measuring microscope at a total magnification of 500 ×. One standard deviation (error bars) is also shown. The dashed line represents the average of all the reported data at the lower magnification, whereas the dotted line is the average of the re-measured ones by authors.

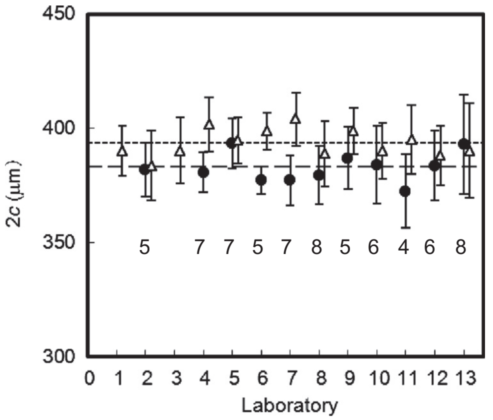

Fig. 4. Crack length, $2c$, of the Si_3N_4 samples measured at the second round robin. Closed circles represent each laboratory's average observed at a total magnification of 400 ×, while open triangles denote those re-measured by authors with a measuring microscope at a total magnification of 500 ×. Number of specimens measured by each laboratory and one standard deviation (error bars) are also shown. The dashed line represents the average of all the reported data, whereas the dotted line is the average of all the re-measured ones by authors.

fact that our well-trained operator had a greater experience in finding the crack tip than each lab's operator may also count for the difference in $2c$. The diagonal sizes reported by each lab are shown in Fig. 5 (closed circle) together with those by the authors (open triangle). The participants' $2a$ exhibited almost the same value to those of the test organizer again and its scatter was not so significant.

Fig. 6 shows that K_{IFR} (closed circle) obtained by using both an objective lens of 40 × and a traveling stage was in the range from 7.5 to 8.4 MPa $m^{1/2}$. The average of all the reported K_{IFR} was 7.8 ± 0.3 MPa $m^{1/2}$(dashed line), which became closer to the values re-measured by authors, 7.5 ± 0.2 MPa $m^{1/2}$ (dotted line). The slight deviation of the average of each lab's K_{IFR} from that of our re-measured ones originated from the minor misreading of crack length by the participants.

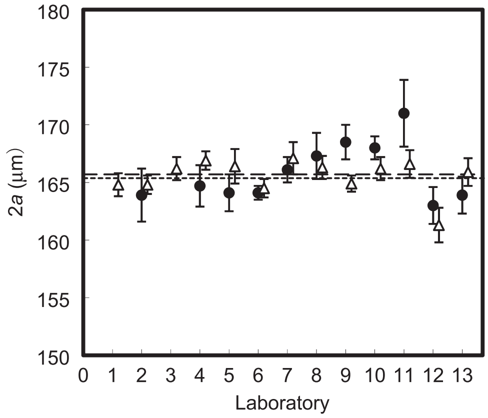

Fig. 5. Diagonal size, $2a$, of the Si_3N_4 samples measured at the second round robin. Closed circles represent each laboratory's average observed at a total magnification of 400 ×, while open triangles denote those re-measured by authors with a measuring microscope at a total magnification of 500 ×. One standard deviation (error bars) is shown. The dashed line represents the average of all the reported data, whereas the dotted line is the average of all the re-measured ones by authors.

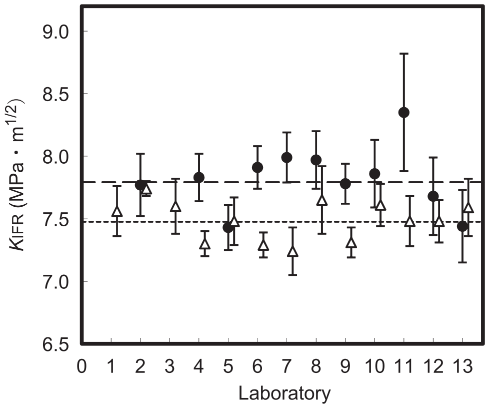

Fig. 6. Results of the second round robin on indentation fracture resistance of the Si_3N_4 samples. Closed circles represent each laboratory's average observed at a total magnification of 400 ×, while open squares denote those re-measured by authors with a measuring microscope at a total magnification of 500 ×. One standard deviation (error bars) is also shown. The dashed line represents the average of all the reported data, whereas the dotted line is the average of the re-measured ones by authors.

These results were analyzed numerically in accordance with the Japanese Industrial Standard Z8402-2 [21] to evaluate the accuracy of measurement methods and results and are shown in Table 1. The repeatability characterizes the variance of the results within each laboratory, that is, the variance of the results obtained by the same operator, with the same equipment in the short period of time. The reproducibility describes the dispersion of the results among the laboratories. It was revealed that the difference between the average of each lab's K_{IFR} and that of our re-measured ones decreased from 9.2% to 4.2% when the magnification of objective lens was increased from 10 × to 40 ×. The standard deviation between labs was diminished as well. Fig. 7 shows the comparison of coefficient of variance (COV) between Si_3N_4 and SiC obtained in our round-robin tests. The reproducibility of the second round robin for Si_3N_4 was almost the same as that for SiC when the

Table 1
Accuracy of the IF method based upon the round robin results according to JIIS Z 8402-2 for the silicon nitrides indented at 196 N [21]. Results of re-measured values by AIST are also included for comparison.

Observer	Magnification of objective lens	Labs	Total indent.	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]
Each lab, 1st	10, 13 or 20	11	69	8.17	0.21	2.6	0.38	4.6
Each lab, 2nd	40	11	68	7.79	0.25	3.2	0.34	4.4
AIST	50	13	101	7.48	0.18	2.4	0.23	3.0

[a] Coefficient of variance.

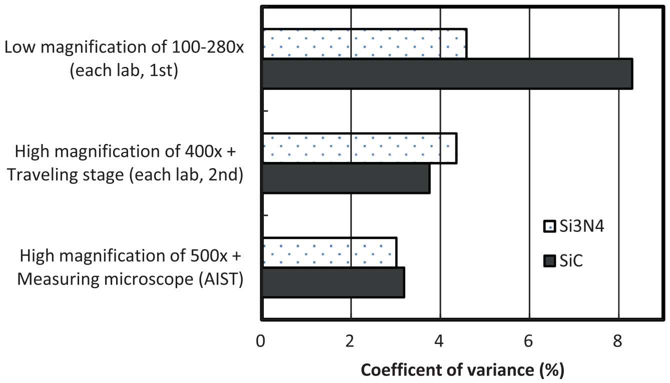

Fig. 7. Comparison of the coefficient of variance between Si_3N_4 and SiC samples measured by each apparatus. The data of the SiC was obtained from the literature [16].

higher magnification of 400 × was used for both materials, indicating the usefulness of our new method to other ceramics besides SiC. The round-robin test which was conducted to internationally-standardize the surface crack flexure (SCF) method for toughness evaluation demonstrated that its reproducibility of K_{IC} of the hipped Si_3N_4 sample was 5.00 ± 0.45 MPa $m^{1/2}$ and its COV = 9% [22]. The reproducibility obtained in our round-robin tests was better than that of the SCF method, suggesting that the IF method with our new technique had the sufficient lever for the international standardization. Thus the reliability of K_{IFR} of silicon nitride could be improved as compared to that of the first round robin by using both the objective lens of 40 × and the traveling stage. The better measurements with the object lens of 40 × can be explained by its fine resolution as discussed in our previous paper [16].

3.3. *Comparison of the improvement in the accuracy of measurements between SiC and Si_3N_4*

Our previous round robin on the K_{IFR} of silicon carbide demonstrated that the accuracy of the measurements was markedly improved, that is, the error in K_{IFR} was reduced from 19.4% to 3.3% by applying our new measuring method [16]. Concurrently, the coefficient of variance of K_{IFR} also decreased dramatically from 8.3% to 3.8% as shown in Fig. 7, which indicated the refinements of precision. By contrast, the improvements in both accuracy and precision of K_{IFR} for the silicon nitride ceramics were moderate as compared to those of SiC. The different behaviors between the two materials can be explained by the visibility of the crack as follows. The crack opening displacement (COD) very close to the crack tip is roughly estimated by the so-called Irwin equation.

$$\delta = \sqrt{(8/\pi)}\cdot K_{I0}/E\cdot\sqrt{x} \qquad (2)$$

where δ is the crack opening displacement, K_{I0} and E are the crack-tip fracture toughness and the Young modulus, respectively, and x is the distance from the crack tip. Young's modulus of Si_3N_4 sample used in this exercise was 284 GPa. K_{IC} obtained by the SEPB method was 7.0 MPa $m^{1/2}$ and the K_{I0} was assumed to be the same as $K_{IC(SEPB)}$ for the rough estimation, which resulted in the value of $\sim 24 \times 10^{-6}$ for K_{I0}/E. By contrast, K_{I0}/E was calculated to be $\sim 5 \times 10^{-6}$ for the silicon carbide specimen in our previous round robin by substituting the $E = 365$ GPa and $K_{I0} = K_{IC(SEPB)} = 1.9$ MPa $m^{1/2}$. Thus δ in the vicinity of the crack tip for Si_3N_4 is estimated more than four times larger than that of SiC. The crack tip fracture toughness, K_{I0} for Si_3N_4 is expected to be much smaller than K_{IC} measured by the SEPB method due to the possible R-curve behavior of the sample. Fünfschilling et al. reported that there is a common crack-tip toughness of $K_{I0} \sim 2.2$ MPa $m^{1/2}$ for three different Si_3N_4 samples [23]. The real K_{I0} for the Si_3N_4 sample in this study may be near to their value. Even in that case, K_{I0}/E is calculated to be $\sim 8 \times 10^{-6}$ for Si_3N_4, which is 60% larger than that of SiC. It is natural to suppose that the visibility of the crack tip is better for Si_3N_4 than SiC due to the wide crack opening of the former ceramics. Accordingly, the misreading of the crack length for silicon nitrides is expected to be not as serious as that for SiC even when the poor optics is employed for the observation.

4. Conclusion

Round-robin tests with 13 laboratories were performed on the indentation fracture resistance, K_{IFR} of silicon nitride ceramics. At the first round robin, both crack length and diagonal size of the indentations at 196 N were measured basically using a microscope equipped with the hardness tester at the low total magnification of 100–280 ×. The crack lengths, $2c$, of the returned samples were re-measured by the authors and compared with the reported values from each participant to clarify the origin of the variation in K_{IFR}. The same indentations were measured with both objective lens of

40 × and traveling stage in the second round robin for better assessment. The following results were obtained.

(1) K_{IFR} varied widely from 7.7 to 8.8 MPa m$^{1/2}$ at the first round robin, while K_{IFR} re-measured by authors was constant and was 7.5 ± 0.2 MPa m$^{1/2}$, indicating that almost identical indentations in size were produced regardless of the different indenters used in each laboratory. The main cause of the inaccurate K_{IFR} of each lab was the misreading of 2*c* ranging from 4 to 30 μm, which could be attributed to the difficulty in finding the exact crack tips due to the poor resolution of the objective lens of 10–20 × .

(2) The misreading of 2*c* by each lab was halved at the second round robin by using both the objective lens of 40 × and the traveling stage, which resulted in the improved accuracy of K_{IFR}.

(3) The refinement in the reliability of K_{IFR} of the Si_3N_4 sample by our new technique was not so dramatic as compared to that of SiC in the previous round robin. The different behaviors between the two materials could be explained by the wider crack opening displacement (COD) for the former than the latter.

Acknowledgments

The authors express sincere thanks to all the participants involved in this round-robin test. The authors also acknowledge the workshop for innovation in reliability of bulk ceramics in the Ceramic Society of Japan.

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