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Ceramics International 39 (2013) 611–617

CERAMICS INTERNATIONAL

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Round robin on indentation fracture resistance of silicon carbide ceramics by using a powerful optical microscope

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Received 19 June 2012; accepted 23 June 2012
Available online 28 June 2012

Abstract

Reproducibility of indentation fracture resistance, K_{IFR} of silicon carbides sintered with B and C was evaluated by a round robin with ten laboratories. When the crack length was measured with an optical microscope at a low magnification of $\sim 100\times$, K_{IFR} varied widely from 3.43 to 4.20 MPa $m^{1/2}$, whereas those obtained by a powerful microscopy with both an objective lens of $40\times$ and a traveling stage exhibited a consistent value of 3.20 ± 0.12 MPa $m^{1/2}$. The wide scatter of K_{IFR} for the former measurements was attributed mainly to the variation in misreading of the crack length. It was revealed that the high resolving power of the objective lens of $40\times$ enabled to find exact crack tips easily, which resulted in the good matching of K_{IFR} between laboratories for the latter case. It was suggested that the observation of indentations with powerful optics was effective for improving the reproducibility of the IF method.

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

1. Introduction

Many small ceramic products and components such as bearing balls and cutting tools are used worldwide [1]. For such applications, it is necessary to evaluate their fracture toughness from real parts themselves. However, conventional toughness evaluation methods are difficult to apply since the sizes of these products are smaller than the test specimens needed in these methods. For example, the length of the test piece must be larger than 18 mm for the single edge-precracked beam (SEPB) [2,3] and the surface flaw in flexure (SCF) methods [4]. The indentation fracture (IF) method has been widely used for determining apparent fracture toughness of ceramics since it has been proposed by Lawn et al. [5]. This method is particularly useful when the sizes of available specimens are limited. Therefore, the IF method is regarded as an alternative technique to measure the fracture toughness of small ceramic parts. However, there has been rigorous arguments that the value measured by the IF method does not represent the real fracture toughness and that the term "indentation fracture resistance, K_{IFR}" should be used when the IF method was applied [6,7]. Thus, the American standard specification for silicon nitride bearing balls adopts the term "indentation fracture resistance, K_{IFR}" for the apparent fracture toughness measured by the IF method [8].

The other serious problem of the IF method is the poor between-laboratory consistency, which was revealed by round-robin tests conducted in order to standardize the indentation fracture test for ceramics (e.g. VAMAS [9–12], etc.). However, almost two decades have passed since the last round-robin tests. Processing of structural ceramics has made a big progress during the decades and a measuring instrument has been refined as well. It is likely that the reproducibility of the IF test is improved as compared with those reported by previous round-robin tests. In our previous study, accuracy of the IF test was checked for silicon nitrides by an international round-robin test with six laboratories [13]. An excellent consistency of K_{IFR} between laboratories was attained when bearing-grade silicon nitrides were used, whereas the scatter

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0272-8842/$36.00
http://dx.doi.org/10.1016/j.ceramint.2012.06.072

of K_{IFR} was increased for another sample with some pores. The severe misreading of crack length was found for the latter sample when observed with a microscope having a low magnification of 100×, which resulted in the wide variation in K_{IFR}. It was revealed that the error in reading the crack length was diminished by using a powerful microscope. However, the consistency of the results obtained by the powerful microscopy was not confirmed statistically due to the limited number of participants who used a microscope with a high magnification.

In this study, mirror-finished samples prepared from commercial silicon carbides were delivered to ten laboratories in Japan to investigate whether the test could provide reliable results. The participants consisted of four universities, four 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 from 100× to 280×. Some labs additionally employed a CCD camera or a measuring microscope with an objective lens of 40× or 50× to obtain high magnifications of 400× and higher. After the measurements by each lab, 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 is well known that slow crack growth dose not occur in SiC densified with B additions since they typically do not leave residual phase along grain boundaries. Then, the re-measured crack lengths were compared directly with those reported values.

In the second round-robin test, all the participants observed the indentations at high magnifications of 400× and 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 effect of the powerful objective lens on the reproducibility of the results was studied and discussed with conjunction of its resolving power.

2. Experimental procedure

2.1. Materials

Silicon carbide ceramics sintered with B and C as sintering additives were purchased from IBIDEN Co. Ltd. (IBICERAM SC-850). The bulk density of the sample was 3.024 g/cm^3 and the relative density was calculated to be 94% by using the theoretical density of 3.217 g/cm^3. Young's modulus obtained by the ultrasonic pulse echo method was 365 GPa. The microstructure observed by optical microscopy is shown in Fig. 1. Many small black dots on the mirror-finished surface of the sample indicated that small pores distributed homogeneously. 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.

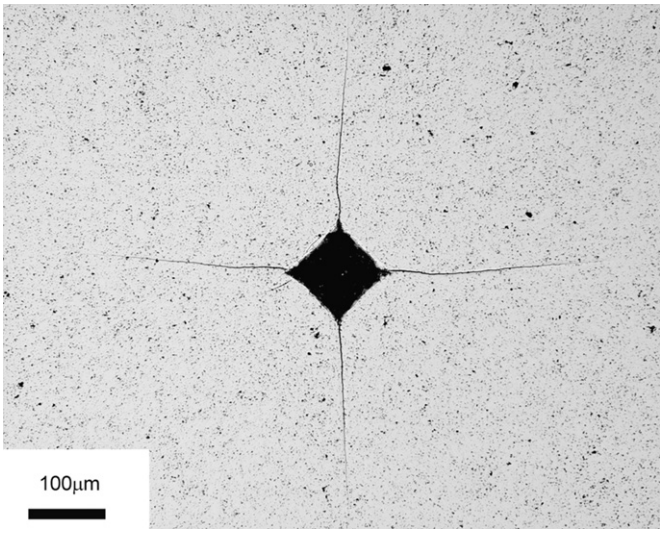

Fig. 1. Optical microscope photo of an indentation at 196 N.

2.2. Test procedure

Vickers indentations were made by each laboratory with a hardness tester. The indentation load was 196 N and the indentation contact time was 15 s. More than eight impressions were made at each laboratory. The lengths of the impression diagonals, 2a, and surface cracks, 2c, were measured immediately after the indentation. Only indentations whose four primary cracks emanated straight forward from each corner were accepted (Fig. 1). Indentations with badly split cracks or with gross chipping were rejected as well as those whose horizontal crack length differed by more than 10% from the vertical one.

Eight labs employed an objective lens of 10× or 13× to measure the size of indentations at the first round robin. Six labs out of eight used a microscope furnished with the tester. They observed impressions directly with an eyepiece of 10×, so that the total magnification was 100× or 130×. Lab no. 9 observed the indentations with the same microscope which equipped with a CCD camera and a monitor, and the total magnification was 280×. Lab no. 4 employed a measuring microscope and the total magnification was 100×. Some labs measured the indentation sizes with another microscope additionally, which provided total magnifications of 400× and higher. Lab nos. 1–4 employed a measuring microscope with an objective lens of 50× (lab nos. 1–3) or 40× (lab no. 4). Their total magnification was 500× or 400×. A microscope with an objective lens of 10× was used by lab nos. 8–10 and the crack tip image captured with a CCD camera was further enlarged on the monitor to obtain the total magnification of 400× or 475×.

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 [14]:

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

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 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 was collected to the test organizer.

All these samples indented by each labs 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 carefully by traveling the 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 acceptable crack morphology. By comparing those data measured by each lab and AIST, it revealed the factor which was dominant, that is, whether the real crack lengths differed themselves due to the variation in hardness tester or the bias of reading the length affected the calculated K_{IFR}.

K_{IFR} reported by lab nos. 1–3 which used a measuring microscope with the magnification of 500 × were similar and were consistent with the re-measured value by the authors. In order to confirm the advantage of the measuring microscope, a round-robin test using a measuring microscope is necessary. However, other labs did not possess the measuring microscope. It was supposed that a microscope equipped with both a powerful objective lens and a traveling stage could be used as a substitute for the measuring microscope, provided that the measurements of length were calibrated properly using a stage micrometer. All participants had such a microscope and could calibrated length measurements correctly. Then, the second round-robin test was conducted using the same samples measured by each lab at the first round robin. The indented samples were sent to each laboratory 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. Almost half of labs observed the impressions directly at a total magnification of 400x with the eyepiece of 10 × . A CCD camera and a monitor were employed by the rest of labs to obtain total magnifications of more than 600 × .

3. Results and discussion

3.1. First round robin using both low and high magnifications

The results of the first round robin for the SiC measured at a low total magnification from 100 × to 280 × were plotted in Fig. 2 (closed square). The ground average of fracture resistance is shown as a dashed line. K_{IFR} varied widely from 3.43 to 4.20 MPa $m^{1/2}$. These results were analyzed numerically in accordance with the Japanese Industrial Standard Z8402-2 [15] 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. The coefficient of variance of reproducibility was 8.3%, which suggested that K_{IFR} obtained by this method was unreliable. The big variation in K_{IFR} among laboratories could be explained by the raw data reported by each laboratory. Table 2 shows that the crack lengths measured by each laboratory varied significantly and were in the range of 574–657 μm. The diagonal sizes were in the range of 133–151 μm (Table 3). According to the law of propagation of errors, the relative errors of K_{IFR}, σ_{KIFR}/K_{IFR} can be estimated by those relative errors of diagonal size, σ_a/a and crack length, σ_c/c as follows:

$$(\sigma_{KIFR}/K_{IFR})^2 = 0.64(\sigma_a/a)^2 + 2.25(\sigma_c/c)^2 \quad (2)$$

The contributions of the first and second terms to the square of relative error of K_{IFR} were calculated roughly as 15% and 85%, respectively, by substituting the relative errors of both diagonal size and crack length, which indicated that the main cause of the wide scatter of K_{IFR} was the dispersion of the crack length.

Measurements at the high magnification using a CCD camera (lab. nos. 8–10) yielded similar K_{IFR} values to

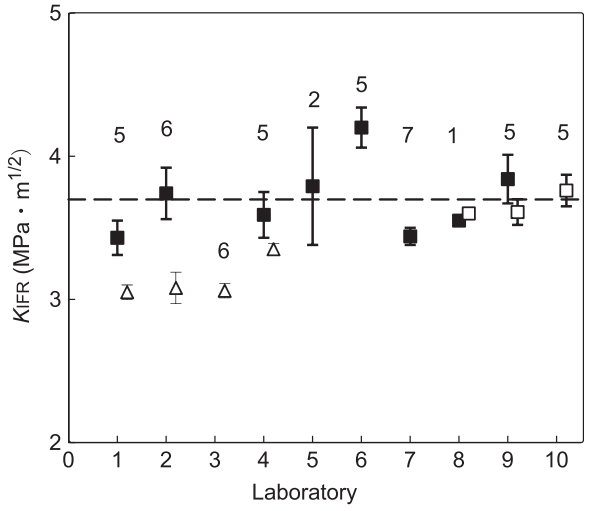

Fig. 2. Results of the first round robin on indentation fracture resistance of the SiC samples. Closed squares represent each laboratory's average observed at a lower magnification of 100 × or 130 × or 280 × (lab no. 9), while open squares denote the average of those obtained with an aid of a CCD camera and the monitor at a total magnification from 400 × to 475 × . Open triangles represent those measured with a measuring microscope at a total magnification of 500 × (lab nos.1–3) or 400 × (lab no.4). 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 at the lower magnification.

Table 1
Accuracy of the IF method based upon the round robin results according to JIIS Z 8402-2 for the silicon carbides indented at 196 N [15]. Results of re-measured values by AIST are also included for comparison.

Observer	Magnification of objective lens	Labs	Total indent.	Average	Repeatability (within-lab)		Reproducibility (between-labs)	
				(MPa $m^{1/2}$)	Std. dev. (MPa $m^{1/2}$)	COV %[a]	Std. dev. (MPa $m^{1/2}$)	COV %[a]
Each lab, 1st	10 or 13	8	36	3.69	0.16	4.3	0.31	8.3
AIST	50	10	58	3.09	0.08	2.5	0.10	3.2
Each lab, 2nd	40	9	43	3.20	0.11	3.4	0.12	3.8

[a]Coefficient of variance.

Table 2
Means and standard deviations data of the crack length, $2c$, for the SiC samples indented at 196 N.

Lab. no.	Lab. readings at low magnification (μm)	Lab. readings at high magnification (μm)	AIST readings with a measuring microscope (μm)	Lab. readings using an objective lens of 40 × and a traveling stage (μm)
1	644.0 ± 13.8	696.4 ± 8.3[a]	708.0 ± 14.2	701.3 ± 17.5
2	601.1 ± 17.2	682.6 ± 9.8[a]	698.1 ± 8.3	–
3	–	690.0 ± 3.2[a]	701.6 ± 6.4	681.1 ± 7.9
4	632.8 ± 16.9	665.4 ± 6.1[b]	698.5 ± 8.4	678.9 ± 9.7
5	597.5 ± 41.0	–	706.9 ± 11.0	661.7 ± 28.0
6	574.3 ± 12.6	–	689.5 ± 7.3	666.4 ± 21.5
7	628.9 ± 6.6	–	687.8 ± 12.3	663.4 ± 13.0
8	656.7	655.0[c]	711.4 ± 8.2	680.5
9	608.4 ± 14.4	634.3 ± 6.9[c]	697.7 ± 18.3	680.6 ± 14.4
10	–	605.8 ± 7.5[c]	686.3 ± 9.2	677.1 ± 8.3
Mean [d]	615.6 ± 27.2	–	697.8 ± 12.7	676.5 ± 17.8

[a]Measured with a measuring microscope with an objective lens of 50 × (total magnification: 500 ×).
[b]Measured with a measuring microscope with an objective lens of 40 × (total magnification: 400 ×).
[c]Measured with an aid of a CCD camera and a monitor at a total magnification from 400 × to 475 ×.
[d]Grand average using all data.

Table 3
Means and standard deviations data of the diagonal size, $2a$, for SiC samples indented at 196 N.

Lab. no.	Lab. readings at low magnification (μm)	Lab. readings at high magnification (μm)	AIST readings with a measuring microscope (μm)	Lab readings using an objective lens of 40 × and a traveling stage (μm)
1	138.6 ± 1.6	138.7 ± 0.6[a]	139.0 ± 0.8	140.6 ± 1.1
2	135.6 ± 2.3	135.1 ± 2.8[a]	139.6 ± 1.4	–
3	–	137.2 ± 2.5[a]	141.3 ± 1.9	137.5 ± 3.4
4	142.3 ± 1.9	143.2 ± 1.1[b]	143.3 ± 1.0	140.4 ± 1.5
5	135.8 ± 1.1	–	139.5 ± 1.3	134.2 ± 2.8
6	144.0 ± 1.8	–	141.1 ± 1.2	136.4 ± 2.6
7	133.3 ± 0.7	–	142.2 ± 1.4	137.7 ± 1.2
8	150.5	152.3[c]	146.5 ± 1.9	144.0
9	143.7 ± 2.1	143.7 ± 2.1[c]	142.4 ± 1.8	143.7 ± 2.1
10	–	138.8 ± 3.5[c]	143.2 ± 1.6	141.8 ± 1.5
Mean[d]	139.2 ± 4.9	–	141.7 ± 2.4	139.3 ± 3.4

[a]Measured with a measuring microscope with an objective lens of 50 × (total magnification: 500 ×).
[b]Measured with a measuring microscope with an objective lens of 40 × (total magnification: 400 ×).
[c]Measured with an aid of a CCD camera and a monitor at a total magnification from 400 × to 475 ×.
[d]Grand average using all data.

those measured at the low magnification (Fig. 2, open squares). The slight difference in K_{IFR} obtained at low and high magnifications could be attributed to the negligible change in both crack length and diagonal size measured at high magnifications (Tables 2 and 3). By contrast, K_{IFR} decreased notably and showed consistent value around

3.06 MPa $m^{1/2}$ when the indentations were measured with the measuring microscope at the high magnification of 500 × (Fig. 2, lab. nos. 1–3, open triangles). The crack lengths measured by this technique were more than 50 μm longer than those obtained at the low magnification and exhibited almost uniform value around ~690 μm (Table 2).

3.2. Comparison with re-measured values by authors using a measuring microscope

K_{IFR} re-measured by authors with a measuring microscope are shown as open triangles in Fig. 3. Almost constant value of 3.09 MPa $m^{1/2}$ was obtained regardless of the difference in the hardness tester used by each laboratory, which was much smaller than those values measured at the low magnification by each participant. The error bars for each data point decreased as compared with those obtained at the low magnification in Fig. 2. These results were analyzed numerically and the accuracy of results is shown in Table 1. It was supposed that the reproducibility of re-measured data by AIST stood for the scatter due to the difference in equipments for making indentations used in each laboratory since the reader of the size of both cracks and diagonals and the microscope were the same. The reproducibility of re-measured data in Table 1 was quite small, ~0.1 MPa $m^{1/2}$, suggesting that almost identical indentations in size were produced by the different hardness testers used in each laboratory. The suggestion could be confirmed directly by our raw data. Tables 2 and 3 demonstrate that both re-measured crack length and diagonal size varied negligibly among the laboratories, which is consistent with our previous round robin on silicon nitride ceramics where the crack lengths re-measured by authors were almost the same for indentations made at different labs [13]. It is likely that the condition of the edge of indenter used in this study varied among the participants, implying that the slight wear of indenter hardly affects the sizes of impressions as was reported by authors previously [16].

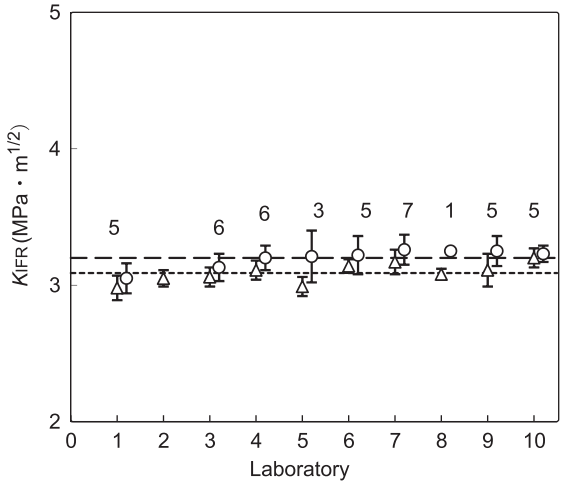

Fig. 3. Results of the second round robin on indentation fracture resistance of the SiC samples measured with an objective lens of 40 × and a traveling stage. Open circles represent each laboratory's average, while open triangles denote the average of re-measured values by authors with a measuring microscope. 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 averages of all the re-measured ones by authors.

The grand average of the diagonal size re-measured by authors was ~142 μm, which was similar to the average of all the participant's values at low magnifications (Table 3). By contrast, the grand average of the crack length re-measured by authors was about ~700 μm, which was more than 100 μm larger than the values reported by lab. nos. 2, 5 and 6 at the low magnification (Table 2). All participants missed the real crack tips when they employed the low magnification, which caused the overestimate of K_{IFR}. The misreading of 2*c* calculated from Table 2 ranged widely from 55 to 115 μm, which was the origin of the wide scatter of K_{IFR} reported by each laboratory using the low magnifications. It was inferred that detecting exact crack tips at the low magnification were difficult and susceptible to the subjectivity of the operators.

Table 2 also shows that the misreading of crack lengths was more than 50 μm even when they were observed by using CCD cameras at the high magnifications (lab. nos. 8–10), indicating that the enlargement of digital image of the crack tips on the monitor did not work as they expected. By contrast, the observations using a measuring microscope with an objective lens of 50 × by lab nos. 1–3 gave crack lengths of ~690 μm, which were similar to those re-measured by authors. It is apparent that using objective lens of high magnification was effective for both finding exact crack tips and reducing the variation in crack length.

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

In order to confirm the accuracy of the measurements using the objective lens with a high magnification of 40 ×, SiC samples were sent back again to each laboratory and the same indentations made at the first round robin were measured by each lab at the second round robin. Fig. 3 shows that K_{IFR} obtained by using both an objective lens of 40 × and a traveling stage (open circles) matched well with the values re-measured by authors and varied little among the laboratories. Table 1 indicated that both repeatability and reproducibility of this method were quite small and were slightly larger than those obtained by authors, which implied that the variation of reading crack length and diagonal size was little. The raw data in Tables 2 and 3 support this suggestion. Table 2 indicates all the crack lengths resided between 661 and 701 μm when measured by this technique. The standard deviation of all 2*c* data was 17.8 μm, which was markedly improved as compared with that of the first round robin at the low magnification and was slightly higher than that of re-measured values by

authors. Thus, it was revealed that the scatter of the crack length could be diminished by using both an objective lens of 40 × and a traveling stage. However, the grand average of 2*c* for this method was about 20 μm shorter than that of re-measured value by authors. It is likely that the small misreading of crack length may be brought about by the bias due to the operators and/or the difference in the magnification of objective lens.

3.4. Effect of the magnification of the objective lens on the accuracy of the measurements

Fig. 4 shows crack tips observed with an optical microscope using objective lenses of three different magnifications, 50 × , 20 × and 10 × . The pictures were taken by a CCD camera with 10 M pixels. The total range of microscope progressively increased with decreasing the magnification of the lens. In order to compare the crack tips in the same magnification, the small region around the crack tip was cut out from the picture obtained at the lower magnification and was enlarged digitally. The very narrow crack tip which located slightly above the white line could be detected clearly when it was observed with the objective lens of 50 × (Fig.4, top). The image of the same crack tip became ambiguous when the magnification of the objective lens decreased to 20 × . In this case, the half of crack length, *c*, may be measured 15 μm shorter than that measured with the objective lens of 50 × . When the magnification of objective lens became 10 × , the crack tip located just above the white line in the former pictures completely disappeared. The image of the crack next to this part became faint, whose length was about 15 μm, so that the operator may misread the half of crack length, *c*, 15–30 μm shorter than that measured with objective lens of 50 × . The difference in crack length, 2*c*, measured with the objective lens of 50 × and 10 × amounted to ~60 μm, which was corresponding roughly to the degree of the misreading of the crack length measured at the first round robin using objective lens of 10 × or 13 × (Table 2).

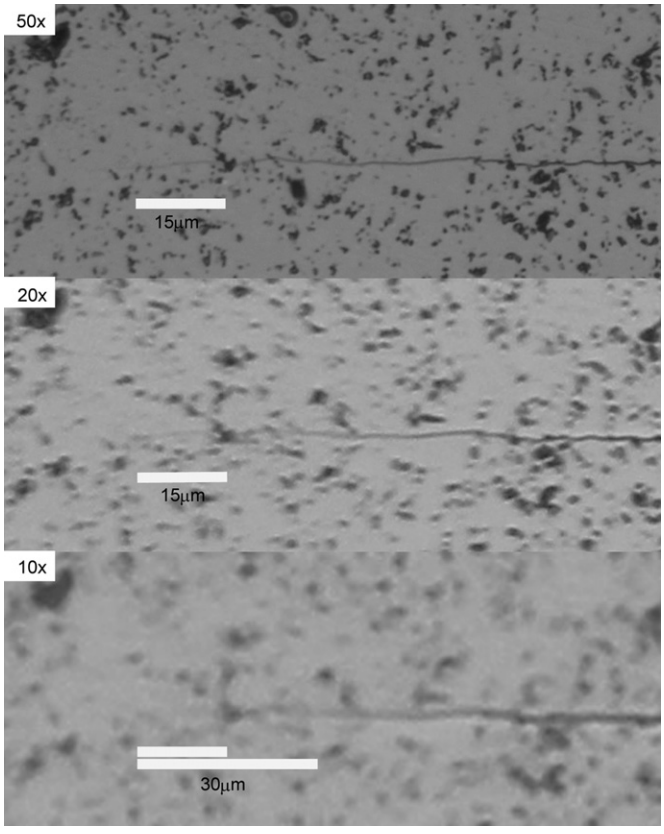

Fig. 4. Examples of crack tips observed by optical microscopy with an objective lens of 50 × , 20 × and 10 × .

It is well known that the resolving power of an optical microscope is determined by the performance of an objective lens as follows:

$$\delta = 0.61\lambda/NA \tag{3}$$

where δ is the two point resolving power, λ is the wave length of the light and *NA* is the numerical aperture of the objective lens. The *NA* of the objective lens of 50 × and 10 × used for Fig. 4 were 0.55 and 0.2, respectively. If we employ green light ($\lambda = 500$ nm), the resolving power for the objective lens of 50 × and 10 × was estimated as 0.55 μm and 1.5 μm, respectively. The origin of the misreading of crack length measured by using the objective lens of 10 × can be explained by its poor resolving power as follows. Usually, observers start searching the crack tip from the corner of the indentation where the crack is wide. The identification of crack becomes progressively difficult as observers approach to the crack tip since the crack opening displacement gradually decreases. When the crack width becomes less than 1.5 μm, the operators cannot identify the crack clearly if the objective lens of 10 × is used because of its limited resolving power. Then the crack with width less than 1.5 μm would be missed and the crack length would be measured shorter. By contrast, the very narrow crack with width of 0.6 μm in the vicinity of the real crack tip can be detected with the objective lens of 50 × . Accordingly, the high resolution image produced by objective lens of 50 × enable to find the real crack tip easier and can give more accurate crack length than that obtained with the objective lens of 10 × . It should be also noted that the enlargement of the CCD-camera image obtained with the objective lens of 10 × on the monitor is not effective for the precise measurements since the resolving power is determined by the performance of the objective lens, which can account for the slight improvement in reading crack length at high magnification of 400 × or 475 × using CCD camera (Table 2, labs no. 8–10).

4. Conclusion

Round-robin tests of indentation fracture resistance, K_{IFR} were conducted with ten laboratories using silicon carbide ceramics as a test material. At the first round robin, the indentations at 196 N were made by each participant and both crack length and diagonal size were measured basically using a microscope equipped with the hardness tester at the low total magnification from 100 × to 280 × . The crack lengths of the returned samples were

re-measured by the authors and compared with the reported values from each participant to clarify the origin of variation in K_{IFR}. The same indentations were measured with both objective lens of 40 × and traveling stage in the second round robin. The following results were obtained:

(1) Quite low reproducibility of K_{IFR}, 3.69 ± 0.31 MPa $m^{1/2}$, was attained for the first round robin. By contrast, the reproducibility of K_{IFR} re-measured by authors was excellent, 3.09 ± 0.10 MPa $m^{1/2}$, indicating that almost identical indentations in size were produced by the different hardness testers used in each laboratory. The wide scatter of the crack length measured at the low magnification by each laboratory was a main cause of the variation of K_{IFR}. The severe misreading of crack length which ranged widely from 55 to 115 μm implied that detecting exact crack tips with poor optics was difficult and susceptible to operator's subjectivity.
(2) The enlargement of the crack image captured by a CCD camera on the monitor did not improve the accuracy of measurements of crack length. By contrast, good agreements were obtained between lab results and those of re-measured by authors when a measuring microscope with an objective lens of 50 × was used.
(3) Each laboratory could measure the crack length more precisely and gave consistent value by using both an objective lens of 40 × and a traveling stage, which resulted in the superior reproducibility of K_{IFR}, 3.20 ± 0.12 MPa $m^{1/2}$.
(4) The usefulness of an objective lens with high magnification in reading crack length was explained by its high resolving power which enabled to detect thinner crack tip than that could be found with an objective lens of low power.

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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