Available online at www.sciencedirect.com

ScienceDirect

Ceramics International 36 (2010) 899–907

CERAMICS INTERNATIONAL

www.elsevier.com/locate/ceramint

Indentation fracture resistance test round robin on silicon nitride ceramics

Hiroyuki Miyazaki *, Yu-ichi Yoshizawa, Kiyoshi Hirao, Tatsuki Ohji

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

Received 26 June 2009; received in revised form 10 July 2009; accepted 18 October 2009
Available online 20 November 2009

Abstract

Reproducibility of indentation fracture resistance of three commercial silicon nitrides including bearing balls was evaluated by an international round robin with six laboratories. The between-laboratory standard deviations for indentations at 196 N on the perfectly mirror-finished surfaces were in the range of 0.2–0.5 MPa $m^{1/2}$, demonstrating an excellent precession of the test results. The scatter in the fracture resistance increased as the indentation load decreased from 196 to 98 N. The errors in measuring crack lengths deduced from the deviation of each laboratory's readings from author's reading for the same indentations tended to increase with a decrease in the magnification of the lab's microscope, which suggested that finding exact crack tips with lower magnification was difficult especially for those samples with insufficiently mirror-finished surfaces indented at 98 N. Observation of indentations at the load of 196 N with powerful optics was advised to ensure the validity of the indentation technique which is used as the quality assessment of Si_3N_4 bearing balls.

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

1. Introduction

The indentation fracture (IF) method has been widely used for determining fracture resistance of ceramics since it has been proposed by Lawn et al. [1]. This method is particularly useful when the sizes of available specimens are limited. Silicon nitride ceramics have been applied to bearing balls due to its outstanding tolerance during the usage in severe conditions [2]. For such applications, it is necessary to evaluate their fracture resistance from real parts themselves. However, conventional toughness evaluation methods such as single edge-precracked beam (SEPB) [3,4] and surface flaw in flexure (SCF) methods [5] are difficult to apply since the sizes of most of the tribological parts such as bearings are smaller than the test specimens needed in these methods. Therefore, the IF method is considered to be an alternative technique to measure the fracture resistance, K_{ifr} of silicon nitride bearing balls and has been adopted in the American standard specification for silicon nitride bearing balls [6].

However, the IF method has not been without detractors. They claimed that the technique should not be used as the formal material specifications since in between-laboratory consistency was poor [7,8], which was revealed by round robin tests conducted in order to standardize the indentation fracture test for ceramics (e.g. VAMAS [9–11], etc. [12]). However, almost two decades have passed since the last round robin tests. Performance of the structural ceramics has made a grade progress during the decades and the measurement equipments have been also refined. It is likely that the accuracy of the IF test is improved as compared with those reported by previous round robin tests. Accordingly, reproducibility of the IF test should be checked especially for bearing-grade silicon nitrides. In this study, mirror-finished samples prepared from three kinds of silicon nitrides including bearing balls were delivered to six laboratories in Ireland, Korea, U.S.A. and Japan (Table 1) to investigate whether the test could provide reliable results. The effect of surface finish of the Si_3N_4 samples on the test accuracy was also studied.

Another important point is that there have been few systematic studies about the origin of large variations in the previous round robin tests [13], although some conjectured that a plausible reason of the scatter among several laboratories was subjectivity of the operators in reading the crack length

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

0272-8842/$36.00
doi:10.1016/j.ceramint.2009.10.027

Table 1
List of participants.

Country	Laboratory
Ireland	University of Limerick
Korea	Korea Institute of Materials Science
U.S.A.	Oak Ridge National Laboratory
Japan	Ceramics Maker
	National Institute of Advanced Industrial Science and Technology (AIST)
	Yokohama National University

[7,9–12]. Because of the industrial need for fracture resistance assessment of small silicon nitride bearing balls, it is important to eliminate the source of scatter by clarifying the origin of measuring errors. Two main origins of variation in K_{ifr} were assumed in this study. The first one is difference in machine which introduces indentations. In this case, real crack lengths themselves are expected to differ among the laboratories. The second one is systematic biases of measurement due to different operators. In order to discern the two factors, authors rechecked all of the returned IF samples since silicon nitride was not susceptible to environmentally assisted postindentation slow clack growth [13–15].

2. Experimental procedures

2.1. Materials

Silicon nitrides from commercial sources were used as test materials. Material identifications and their suppliers are listed in Table 2. The TSN03 sample was bearing-grade silicon nitrides, and real bearing balls were also used. These were processed by HIP to attain high densification and high elastic modulus, whereas other materials expressed lower values of both density and Young's modulus. The microstructures observed by scanning electron microscopy (SEM) are shown in Fig. 1. Considerable variation in the microstructures was observed for the three samples. The TSN03 sample consisted of fine and uniform grains, which was typical microstructure for bearing-grade silicon nitrides [16]. Relatively larger amounts of intergranular glassy phase were found among needle-like grains in the SN1 sample. Much larger grains (>6 μm) existed occasionally in the SN220 sample.

Rectangular specimens with dimensions of 4 mm × 3 mm × 44 mm were machined from the sintered samples. The larger 4 mm × 44 mm surface was polished to a mirror finish for indentations. In the case of the TSN03-bearing balls, disc samples with 3 mm thick were prepared from 3/8 in. bearing balls (Fig. 2), followed by polishing of one side of the desk to an optical finish. The conditions of mirror-finished surfaces were progressively more difficult to read crack lengths depending on the microstructures. The surface of TSN03-bearing-grade sample was mirror-finished perfectly without residual pores, which was easiest to read crack lengths (Fig. 3). That of the SN1 sample was also fine, but with minor pores. By contrast, the SN220 possessed many residual pores and the coarsest microstructures, making it challenging to read exact crack tip positions [17]. The test with the SN220 was conducted to reveal the lowest level of accuracy in the bad condition. The test was also expected to suggest the origin of errors clearly, which will demonstrate necessary conditions for accurate measurements.

2.2. Test procedure

Vickers indentations were made by each laboratory with a hardness tester. The indentation contact time was 15 s. Indentation loads of 98, 196 and 294 N were chosen to vary the crack size. However, half of labs could not indent at 294 N because of the limited capacity of their testers. Indentations in the TSN03-bearing ball samples were performed only at 196 N. Eight impressions were made at each load. Measuring conditions for the indentations at each labs were summarized in Table 3. Most labs employed bright field images, while lab No. 5 used the Nomarski technique. Total magnifications of half of labs were as low as 100× since their microscopes were furnished with the tester. Labs No. 5 and 6 used the metallurgical microscope or the CCD camera equipped with the microscope to increase the magnification 200× and over. The best resolution was obtained by the measuring microscope (lab No. 1), by which the crack tips were detected at the highest magnification of 500× and the spacing between the tips was measured preciously by traveling the stage with a readout resolution of 1 μm. 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. 3). 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.

The indentation fracture resistance, K_{ifr}, was determined from the as-indented crack lengths by the Niihara's equation for the median crack system as follows [18]:

$$K_{\mathrm{ifr}} = 0.0309\left(\frac{E}{H}\right)^{2/5} Pc^{-3/2} \qquad (1)$$

Table 2
Commercially available silicon nitrides used in this study.

Material identification	Supplier name	Processing	Density (g/cm^3)	Young's modulus (GPa)
TSN03	Toshiba Materials	HIP	3.23	305
TSN03-bearing ball	Toshiba Materials	HIP	3.23	305
SN1	Japan Fine Ceramic Center	GPS	3.20	284
SN220	Kyocera	Sintered	3.20	289

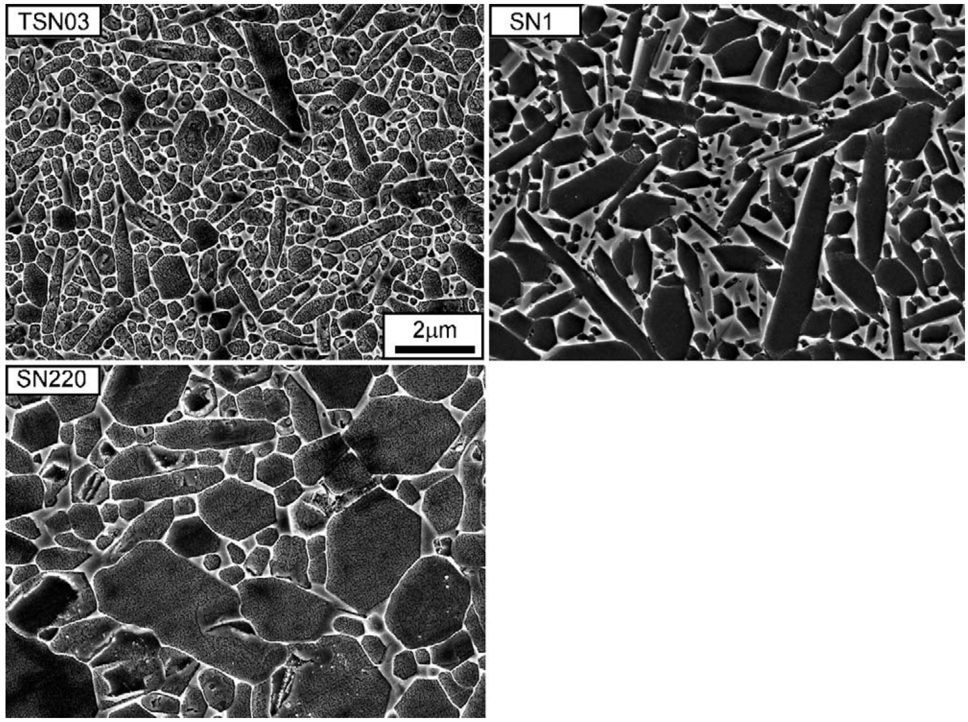

Fig. 1. SEM micrographs of the polished and etched surfaces of silicon nitride ceramics.

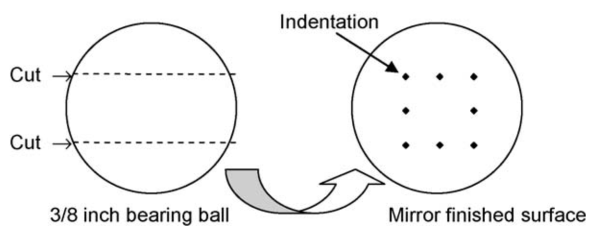

Fig. 2. Schematic illustration of the preparation of a disc sample from a real silicon nitride bearing ball for the indentation fracture test.

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 listed in Table 2 was used. K_{ifr} was calculated for each indentation using the hardness value obtained for each impression. Although the ratios of crack length to diagonal size were smaller than 2.5 for some indentations at 98 N, the crack systems were directly observed to be median/radial ones by a serial sectioning technique or a decoration method [19,20]. Then, the above equation was applied for those indentations. Those calculated K_{ifr} together with the raw data were collected to the test organizer.

All these samples indented by each labs except lab No. 3 were also retuned to author's laboratory (AIST, No. 1) to re-measure the sizes of indentations. The author's data of lab No. 1 was also rechecked by a different operator. In some cases, the numbers of lab's indentations were slightly different from those rechecked by AIST due to the subjective judgments of acceptable crack morphology. By comparing those data measured by each labs and AIST, it was revealed which factor was dominant, that is, whether the real crack lengths themselves differed due to the variation in hardness machine or the bias of reading the length affected the variation in calculated K_{ifr}.

3. Results and discussion

3.1. TSN03 silicon nitrides and bearing balls

The results of round robin for the TSN03 measured at 98, 196 and 294 N were plotted separately in Fig. 4. The ground average of fracture resistance is shown as a dashed line. Excellent coincidences between laboratories were obtained excluding a minor variation in K_{ifr} for indentations at 98 N. Re-measured values by authors were also shown as open circles, which were almost identical to each lab's values. Fig. 5 shows the results for the TSN03-bearing balls measured at an indentation load of 196 N, in which a remarkable coincidence between laboratories was observed, as well as the high rate of acceptable indentations to total indentations.

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 Tables 4 and 5. The repeatability characterizes the variance of

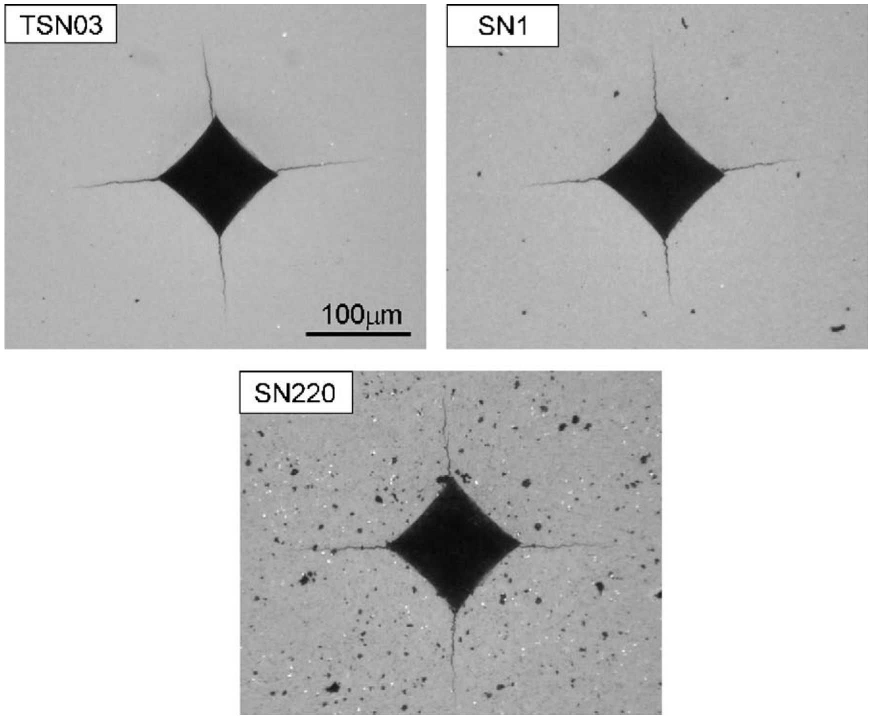

Fig. 3. Optical microscope photos of indentations at 98 N.

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 is 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. Dissimilarity between the reproducibility of all of the reported data by each labs and that of re-measured ones reflects the effects of the bias in reading the lengths since the observed indentations were the same. The reproducibility for re-measured data in Tables 4 and 5 were quite small, $\sim$0.2 MPa m$^{1/2}$, indicating that almost identical indentations in size were produced by the different hardness testers used in each laboratory. The differences in reproducibility were also negligible between the results by each labs and re-measured data by authors, which revealed that the bias in reading crack length and diagonal size was also little. All these features could be confirmed directly by comparing the raw data of both crack and diagonal sizes measured by each lab and re-measured by authors. Table 6 presents the raw data at 98 N for an example, where the differences in crack size between labs and AIST reading were less than 10 μm. Good matches in the raw data were also observed for the indentations at loads of 196 and 294 N. Accordingly, the excellent consistency in $K_{\rm ifr}$ for the TSN03 silicon nitrides was brought about by both the small bias in reading sizes of indentations and the homogeneity of indentation's sizes.

3.2. SN1 silicon nitrides

The data reported by lab No. 2 was written erroneously on the results sheets, so that they were rejected, but the rechecked data using the samples returned from lab No. 2 was included for the analysis. Agreements in $K_{\rm ifr}$ between laboratories were also fine for indentations in the SN1 samples at both 196 and 294 N (Fig. 6), while the variation of

Table 3
Experimental conditions for measuring both diagonal size and crack length.

Laboratory	Microscope	Mode of viewing	Magnification		
			Eyepiece	Objective	Total
1	Measuring microscope	Bright field	10	50	500
2	One equipped with the tester	Bright field	10	10	100
3	One equipped with the tester	Bright field	10	10	100
4	One equipped with the tester	Bright field	10	10	100
5	Metallurgical microscope	Nomarski	10	20	200
6	One equipped with the tester + CCD camera	Bright field	Microscope: 100 CCD camera: 2.8		280

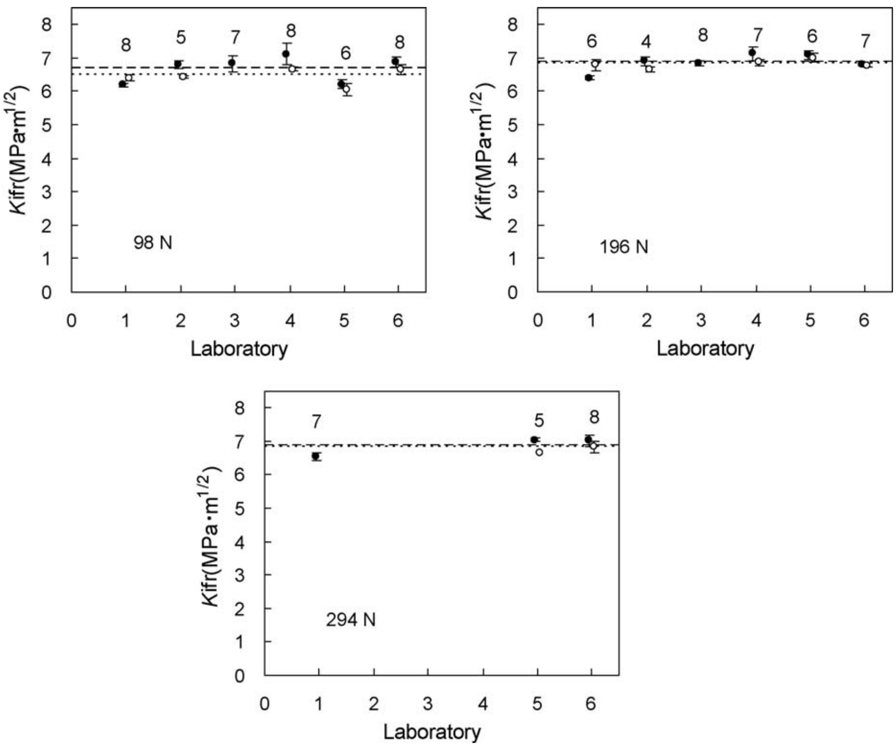

Fig. 4. Results of the round robin on indentation fracture resistance for the TSN03 samples. Closed circles represent each laboratory's average, while open circles denote the average of re-measured values by authors. Number of specimens measured by each laboratory and one standard deviation (error bars) are also shown. The dashed lines represent the averages of all the reported data, whereas the dotted lines are the averages of all the re-measured ones by authors.

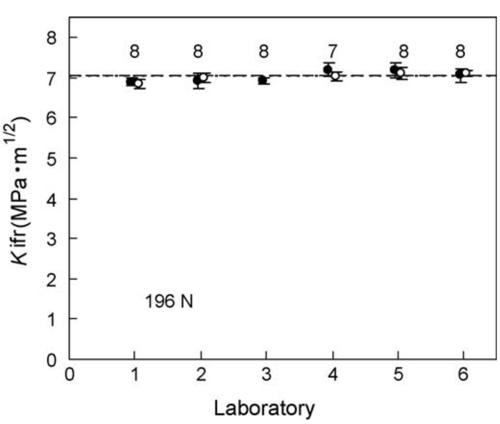

Fig. 5. Results of the round robin on indentation fracture resistance for the TSN03-bearing ball samples. Closed circles represent each laboratory's average, while open circles denote the average of re-measured values by authors. Number of specimens measured by each laboratory and one standard deviation (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.

Table 4
Accuracy of the IF method based upon the round robin results according to JIIS Z 8402-2 for the TSN03 silicon nitrides [21]. Results of re-measured values by AIST are also included for comparison.

Observer	Indentation load (N)	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	98	6	42	6.68	0.19	2.9	0.43	6.4
Each lab	196	6	38	6.87	0.12	1.8	0.28	4.1
Each lab	294	3	20	6.86	0.13	2.0	0.32	4.7
AIST	98	5	35	6.50	0.11	1.6	0.23	3.5
AIST	196	5	40	6.83	0.11	1.6	0.17	2.4
AIST	294	2	8	6.79	0.19	2.9	0.20	2.9

[a] Coefficient of variance.

Table 5
Accuracy of the IF method based upon the round robin results according to JIIS Z 8402-2 for the TSN03 silicon nitride bearing balls [21]. Results of re-measured values are also included for comparison.

Observer	Indentation load (N)	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	196	6	47	7.03	0.16	2.2	0.21	2.9
AIST	196	5	38	7.02	0.12	1.7	0.15	2.2

[a] Coefficient of variance.

Table 6
Means and standard deviations data of the diagonal size and crack length for the TSN03 samples indented at 98 N.

Laboratory	Diagonal size, $2a$ (μm)		Crack length, $2c$ (μm)	
	Lab. readings	AIST readings	Lab. readings	AIST readings
1	109.6 ± 0.8	110.8 ± 0.8	276.6 ± 1.7	269.3 ± 2.7
2	110.8 ± 0.8	108.8 ± 0.5	261.4 ± 2.4	268.7 ± 1.0
3	111.6 ± 2.1	–	261.8 ± 5.3	–
4	111.2 ± 1.4	109.6 ± 0.4	254.2 ± 6.6	263.1 ± 1.7
5	109.7 ± 0.5	109.2 ± 0.3	275.8 ± 3.4	280.2 ± 6.0
6	112.3 ± 1.7	109.3 ± 0.4	261.2 ± 4.5	263.2 ± 4.1

results at 98 N became larger than that of the TSN03 sample. The behavior was also apparent in the quantitative assessment of accuracy of the test for the SN1 in Table 7, where the standard deviation of between laboratories exhibited relatively larger value of 0.6 MPa $m^{1/2}$ when the indentations at 98 N were observed by each laboratories. Fig. 6 also shows that the differences in $K_{\rm ifr}$ between each laboratory and authors were mostly negligible with the exception of the data of laboratory No. 4 at 98 N. Thus, it is expected that the between-laboratory variability in crack length measurements was little. Almost constant re-measured $K_{\rm ifr}$ at each load suggested that the true crack length and diagonal size did not differ significantly among those indentations made by different laboratories with different equipments. The excep-

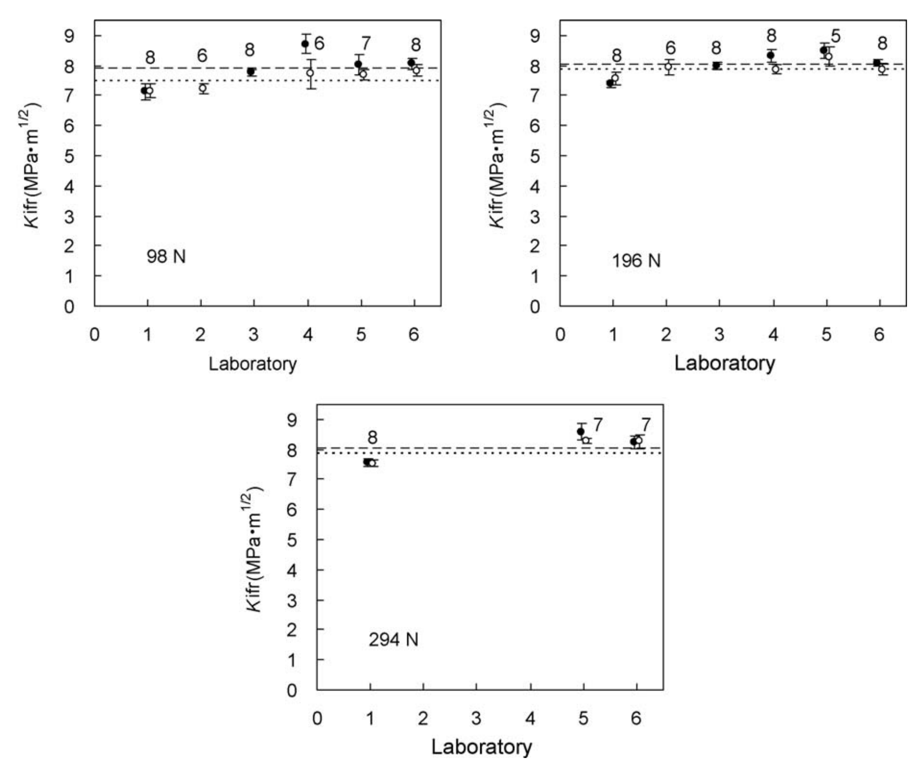

Fig. 6. Results of the round robin on indentation fracture resistance for the SN1 samples. Closed circles represent each laboratory's average, while open circles denote the average of re-measured values by authors. Number of specimens measured by each laboratory and one standard deviation (error bars) are also shown. The dashed lines represent the averages of all the reported data, whereas the dotted lines are the averages of all the re-measured ones by authors.

Table 7
Accuracy of the IF method based upon the round robin results according to JIIS Z 8402-2 for the SN1 silicon nitrides [21]. Results of re-measured values by AIST are also included for comparison.

Observer	Indentation load (N)	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	98	5	37	7.90	0.25	3.1	0.60	7.6
Each lab	196	5	37	8.02	0.17	2.1	0.45	5.6
Each lab	294	3	22	8.11	0.20	2.5	0.56	6.9
AIST	98	4	27	7.47	0.20	2.6	0.39	5.3
AIST	196	5	37	7.88	0.22	2.8	0.31	4.0
AIST	294	3	21	7.98	0.17	2.2	0.47	5.9

[a] Coefficient of variance.

Table 8
Means and standard deviations data of the diagonal size and crack length for the SN1 samples indented at 98 N.

Laboratory	Diagonal size, $2a$ (μm)		Crack length, $2c$ (μm)	
	Lab. readings	AIST readings	Lab. readings	AIST readings
1	115.6 ± 0.8	115.4 ± 1.3	254.3 ± 6.7	253.6 ± 5.5
2	–	115.8 ± 0.6	–	252.0 ± 3.8
3	116.8 ± 1.1	–	241.2 ± 2.2	–
4	118.0 ± 1.1	114.9 ± 0.7	225.0 ± 6.2	240.8 ± 9.8
5	114.9 ± 1.1	115.8 ± 0.3	234.0 ± 5.5	241.9 ± 4.1
6	119.7 ± 0.7	114.9 ± 0.7	238.7 ± 3.6	238.1 ± 4.2

tionally small crack length were reported by laboratory No. 4 for the indentations at 98 N as seen in Table 8, which was the main cause of the abrupt deviation of K_{ifr} for this laboratory at 98 N in Fig. 6.

3.3. SN220 silicon nitrides

The SN220 was rather challenging due to the coarse microstructure and the residual pores as described above. Three

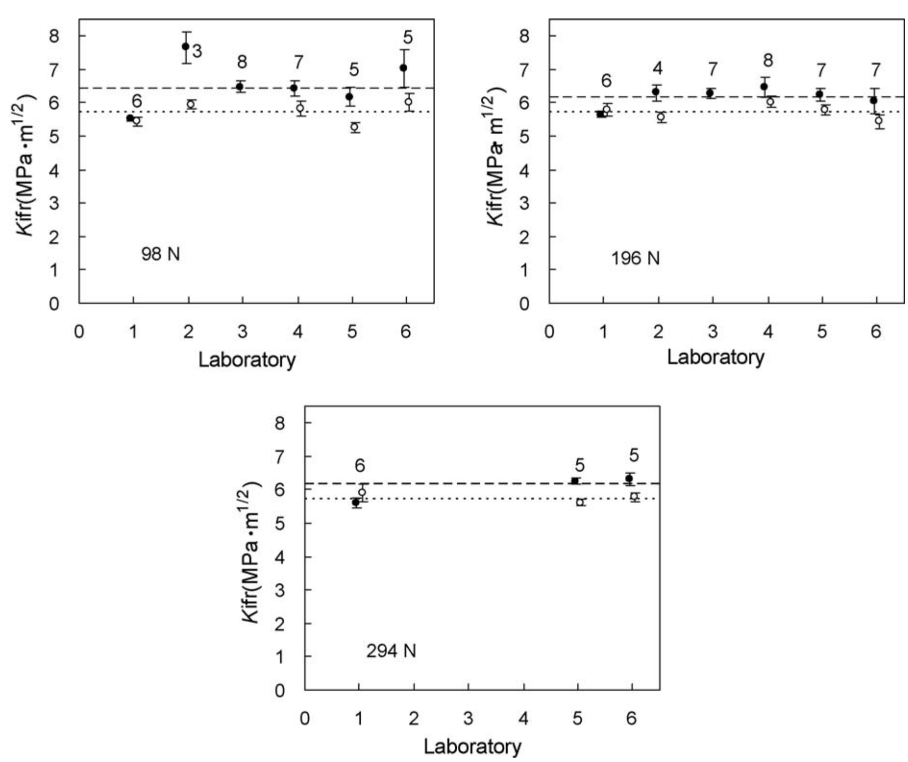

Fig. 7. Results of the round robin on indentation fracture resistance for the SN220 samples. Closed circles represent each laboratory's average, while open circles denote the average of re-measured values by authors. Number of specimens measured by each laboratory and one standard deviation (error bars) are also shown. The dashed lines represent the averages of all the reported data, whereas the dotted lines are the averages of all the re-measured ones by authors.

Table 9
Accuracy of the IF method based upon the round robin results according to JIIS Z 8402-2 for the SN220 silicon nitrides [21]. Results of re-measured values by AIST are also included for comparison.

Observer	Indentation load (N)	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	98	6	34	6.44	0.30	4.7	0.70	10.9
Each lab	196	6	39	6.18	0.25	4.0	0.36	5.8
Each lab	294	3	16	6.04	0.15	2.5	0.42	7.0
AIST	98	5	37	5.72	0.18	3.2	0.37	6.5
AIST	196	5	35	5.73	0.17	3.0	0.28	4.9
AIST	294	3	19	5.75	0.17	2.9	0.21	3.6

[a] Coefficient of variance.

Table 10
Means and standard deviations data of the diagonal size and crack length for the SN220 samples indented at 98 N.

Laboratory	Diagonal size, 2a (μm)		Crack length, 2c (μm)	
	Lab. readings	AIST readings	Lab. readings	AIST readings
1	121.0 ± 1.1	120.2 ± 0.7	310.7 ± 3.2	312.3 ± 4.2
2	122.4 ± 0.9	119.8 ± 1.4	251.3 ± 11.8	293.1 ± 5.2
3	121.4 ± 1.1	–	279.5 ± 5.4	–
4	119.7 ± 1.7	118.4 ± 0.6	278.8 ± 5.6	295.8 ± 7.3
5	119.2 ± 1.0	118.1 ± 0.4	285.7 ± 8.5	316.6 ± 5.4
6	125.8 ± 0.9	117.8 ± 0.9	270.4 ± 14.1	288.9 ± 7.3

plots in Fig. 7 show the test results for the SN220 measured at loads of 98, 196 and 294 N. The worst outcome in this study was obtained at an indentation load of 98 N, where the considerable between-laboratory inconsistency was evident. The reproducibility of the test by each laboratories marked the largest value of 0.7 MPa m$^{1/2}$ at this load (Table 9). In Fig. 7, obvious discrepancies between the grand averages of all K_{ifr} obtained by each laboratory (dashed line) and those re-measured by AIST (dotted line) also appeared. Table 9 also shows that the average of re-measured K_{ifr} was independent on the indentation load, whereas the mean of reported data by each laboratory decreased with increasing the load, gradually approaching the re-measured values. The apparent tendency of decreasing calculated fracture resistance with an increment in indentation load suggested that the measurements by each laboratory were inaccurate since fracture resistance never decreases with increasing crack lengths as the load increases. The raw date at 98 N in Table 10 shows that the crack lengths measured by each laboratory were 20–40 μm smaller than those re-measured by authors, indicating that most of participants missed the real crack tips due to the obstacles such as pores. It is supposed that the contribution of the miss reading of crack length to the K_{ifr} should be decreased as the size of indentation becomes larger, which could account for the apparent decrease in the average of K_{ifr} observed by each laboratory. By contrast, the reproducibility of re-measured K_{ifr} was less than 0.4 MPa m$^{1/2}$ regardless of the indentation loads (Table 9), suggesting that the contribution of the difference in hardness tester to the variation was not so significant for this case as well. Therefore, the worst results among the three materials could be reasonably explained by the difficulties in finding the crack tips in the bad surface condition with residual pores.

The main characteristics of the bearing-grade silicon nitrides are fine and uniform microstructures and superior resistant against wear [16], which enable to create a mirror-finished surface easily. Therefore, it is sure that the accuracy of IF test for bearing-grade silicon nitrides would never fall bellow such a low level observed in the SN220 provided proper polishing is made.

3.4. Effect of the magnification of the microscope on the accuracy of the measurements

The relative differences between the averages of cracks length measured by each laboratory, 2c and those re-measured by authors, 2c* for the same indentations were plotted in Fig. 8

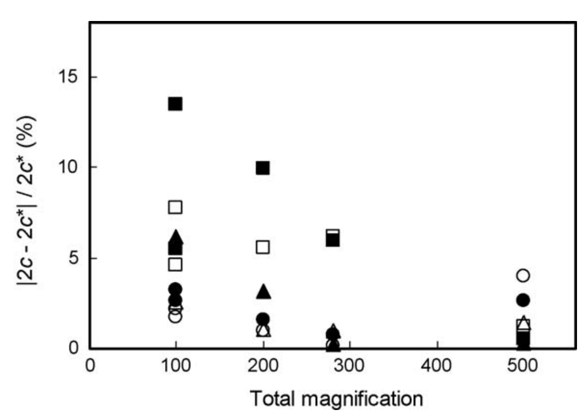

Fig. 8. Relative measuring error for crack length versus magnification of microscope used in each laboratory. The difference between the averages of cracks length measured by each laboratories, 2c and those of re-measured by authors, 2c* were divided by 2c* to estimate the relative error in measuring crack length. Closed marks represent the data for indentations at 98 N, while open ones indicate those at 196 N: circle, TSN03; triangle, SN1; square, SN220.

as a function of the total magnification used for the observation by each laboratory. It was found that the relative measuring errors tented to decrease with the magnification for the SN220 (square) and SN1 (triangle), while those for the TSN03 (circle) did not. Most of the errors for indentations at 98 N (closed marks) were larger than those at 196 N (open marks). The results clearly demonstrated that finding the exact crack tip was difficult with the optics furnished with their hardness tester, especially when the samples without perfect mirror-finished surfaces were indented at the lower load. Indentation load of 196 N and using more powerful microscopes were recommended to avoid inadequate measurements of crack lengths.

4. Conclusion

An international round robin test for evaluating indentation fracture resistance, K_{ifr} was conducted with six laboratories from Ireland, Korea, U.S.A. and Japan. The three materials used in the test were progressively more difficult to measure by the IF method. The TSN03 HIPed silicon nitride samples possessed perfect mirror-finished surfaces which was beneficial to read the crack tip positions. The surfaces of the SN1 gas-pressure sintered samples were also fine but minor pores existed. The SN220 samples were most challenging for detecting the exact crack tips since many residual pores were left on the surfaces beside the coarsest microstructure. 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. The following results were obtained.

(1) Quite low between-laboratory standard deviations as low as 0.2–0.4 MPa $m^{1/2}$ was attained for the bearing-grade silicon nitrides and the disc samples cut from bearing balls, demonstrating the superior between-lab consistency, or reproducibility of the test results.
(2) Agreements between labs results were also excellent for the SN1 samples with an exception of indentations at 98 N.
(3) Severe miss reading of crack length as much as 20–40 μm was found for the SN220 samples, which rendered the trueness of K_{ifr} calculated by each laboratory worst in this study. The deviation of K_{ifr} became evident for those labs which employed the microscope with lower magnification of 100×.
(4) It is presumed that the accuracy of the IF results for other bearing-grade silicon nitrides would be acceptable if the magnification of microscope higher than 200× and indentation load of 196 N were employed since a good optically finished surface was available owing to their fine and uniform microstructures and absence of residual pores.

Acknowledgements

The authors express sincere thanks to all the participants involved in this round robin test: Prof. S. Hampshire, University of Limerick (Ireland); Dr. H-D. Kim, Korea Institute of Materials Science (Korea); Dr. H.T. Lin, Oak Ridge National Laboratory (U.S.A.); Prof. J. Tatami, Yokohama National University (Japan). This work has been supported by METI, Japan, as part of the international standardization project of test methods for rolling contact fatigue and fracture resistance of ceramics for ball bearings.

References

[1] 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.
[2] K. Komeya, Material development and wear applications of Si_3N_4 ceramics, Ceram. Trans. 133 (2002) 3–16.
[3] 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.
[4] Testing methods for fracture toughness of fine ceramics, Japanese Industrial Standard, JIS R 1607, 1995.
[5] 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.
[6] Standard specification for silicon nitride bearing balls, ASTM F 2094-03a, 2003.
[7] G.D. Quinn, Fracture toughness of ceramics by the Vickers indentation crack length method: a critical review, Ceram. Eng. Sci. Proc. 27 (2006).
[8] G.D. Quinn, R.C. Bradt, On the Vickers indentation fracture toughness test, J. Am. Ceram. Soc. 90 (2007) 673–680.
[9] 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.
[10] 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.
[11] 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.
[12] Report of preliminary investigation for standardization of fine ceramics, Japanese Fine Ceramics Association, Japan, 1998.
[13] H. Miyazaki, H. Hyuga, Y. Yoshizawa, K. Hirao, T. Ohji, Study of the factors affecting the lengths of surface cracks in silicon nitride introduced by Vickers indentation, Ceram. Eng. Sci. Proc. 28 (2007) 391–398.
[14] K.D. McHenry, T. Yonushonis, R.E. Tressler, Low-temperature subcritical crack growth in SiC and Si_3N_4, J. Am. Ceram. Soc. 59 (1976) 262–263.
[15] A. Bhatnagar, M.J. Hoffman, R.H. Dauskardt, Fracture and subcritical crack-growth behavior of Y–Si–Al–O–N glasses and Si_3N_4 ceramics, J. Am. Ceram. Soc. 83 (2000) 585–596.
[16] H.I. Burrier, Optimizing the structure and properties of silicon nitride for rolling contact bearing performance, Tribol. Trans. 39 (1996) 276–285.
[17] C.B. Ponton, R.D. Rawlings, Vickers indentation fracture toughness test. Part 2. Application and critical evaluation of standardized indentation toughness equations, Mater. Sci. Technol. 5 (1989) 961–976.
[18] 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.
[19] H. Miyazaki, H. Hyuga, Y. Yoshizawa, K. Hirao, T. Ohji, Crack profiles under a Vickers indent in silicon nitride ceramics with various microstructures, Ceram. Int. 36 (2010) 173–179.
[20] T. Lube, Indentation crack profiles in silicon nitride, J. Eur. Ceram. Soc. 21 (2001) 211–218.
[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.