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Indentation fracture resistance test round robin on silicon nitride ceramics

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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

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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]:

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

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