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The Effect of Indentation Load, Tilted Angle, and the Amount of Surface Removal to the Fracture Toughness of Silicon Nitride Measured by the Surface Crack in Flexure (SCF) Test Method

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Abstract. The surface crack in flexure (SCF) is a method for the evaluation of the fracture toughness of advanced ceramics. Conventionally is practiced by using a Knoop indenter to make a very small precrack. Removal on indent and the plastically deformed zone is required before the fracture test. The purpose of this removal is to eliminate residual stresses under the Knoop impression and to obtain a semi elliptical precrack shape. In this work the influence of the variation of several experimental parameters is investigated. Fracture toughness values by the SCF method are compared with those measured using the SEVNB (single edge V-notched beam) method. The material chosen for this purpose was gas pressured sintered silicon nitride (Si_3N_4) containing 3wt.%Al_2O_3 and 3 wt.%Y_2O_3 (SL200B, Ceram Tec, Plochingen, Germany). The varied parameters were indentation load, orientation of the indentation crack with respect to the bending axis and the amount of material removed from the surface. Additional investigations were performed to determine the crack geometry for various indentation loads. A procedure was developed to facilitate the location and measurement of the precrack size on the fracture surfaces. The effect of measurement accuracy of the results is evaluated. The fracture toughness of specimens with a surface removal in the range suggested from ASTM C 1421 were found to agree with the results obtained from SEVNB. A surface removal below the recommendation resulted in low values of fracture toughness. Increasing the amount of surface removal moderately was found to still fit with results obtained from SEVNB. Surface removal of much more from the recommended amount leads to failure from natural flaws. Observations from serial sectioning revealed that precracks obtained from HK20 and HK30 have an irregular precrack shape with large lateral cracks. It is suggested to use HK5 and HK10 to produce semi-elliptical surface cracks.

1. Introduction

The surface crack in flexure (SCF) is a method for the evaluation of the fracture toughness of advanced ceramics also known as controlled flaw method. Despite this good track record, the SCF method still has the drawbacks. Previous research informed that with serial-sectioning experiments beneath Knoop indentations, observed that a second set of lateral cracks developed very deep beneath the indentation at indentation loads above 98 N [1]. Quinn and Lloyd [2] also detected deeper than expected lateral cracks in a particular machinable glass-ceramic specimen. The oversized lateral crack

was estimated to have interfered with the median crack to the extent that fracture toughness [3,4]. To overcome the lateral crack problem Quinn [4] suggested the lateral cracks may be eliminated by hand grinding or polishing off 7–10 times the indentation depth. The requisite amount can be checked by monitoring the hand-ground tensile surface before specimen fracture. This technique is not convenient to conduct since we need to check regularly in the time of grinding or polishing. Regarding determination the indentation load for the precrack, the standard C 1421 only inform that the determination basic on class of materials *i.e.* approximately 10 to 20 N are suitable for very brittle ceramic, 25 to 50 N for medium tough ceramic, and 50 to 100 N for very tough ceramic[5]. This procedure is not having any significant related with lateral crack problem.
It was reported on previous publications [6,7,8] for silicon nitride in some circumstances, the determination of precrack size was difficult to detect. Park e*t al*. investigated the effect of crosshead speed on the fracture toughness of Si_3N_4. A slow crosshead speed was applied in order to yield slow crack growth (SCG) resulting "halo" region to appear. With appearance of halo region the measurement of the precrack size will be easier to conduct. Unfortunately the halo region induced by SCG did not appear in the Si_3N_4 due to critical crack size at fracture is equal to the initial precrack size induced by indentation [9].
Serial sectioning is introduced in this research to overcome the problem of lateral crack and difficulties in determination of precrack size. It is included also in this work to investigate the influence of the variation of several experimental parameters on fracture toughness by using the SCF method. The results are compared with those measured using the SEVNB (single edge V-notched beam) method [10].

2. Experimental

The material chosen was silicon nitride produced by Ceram Tec (Plochingen, Germany) under the name SL200 B (Table 1). It is a gas pressure sintered ceramic containing ~3 wt.% Al_2O_3 and ~3 wt.% Y_2O_3. This material was a silicon nitride reference material [11]

Table 1. Material Properties

Density	3190±9 kg/m^3
Young modulus	303± 1.3 (bulk), 307± 2(skin)GPa
Hardness HV1, HV 5, HV10	1452, 1461, 1446

SEVNB, the fracture toughness was conducted based on preparation of bar test pieces in which a sharp-tipped notch is machined. The reciprocating razor blade and diamond paste was used for honing the test piece. Under well controlled condition, a notch-tip radius in the range of 1 µm to 20 µm was be obtained for valid condition regarding the standard. The bar test pieces of rectangular with cross section size of 3 mm x 4 mm and length of ≥ 4.5 mm was prepared for these purpose. The test piece in the jig on its 3 mm width face with the V-notch face resting on the outer-support roller. The four-point flexure was employed in this research. The loading rate was applied at 0.5 mm/min. The depth of V-notch was measured by observing the fracture surface using a microscope. Fracture toughness was computed in accordance with ISO/FDIS 23146 and the value was compared to the one that obtained from SCF method.
SCF, The bend specimen for this purpose was prepared with cross section size of 3 mm x 4 mm and length of ≥ 4.5 mm, or halves of previously tested strength specimens. In order to obtain fine precrack, preliminary research with serial sectioning was carried out on 4 variations of Knoop indentation load namely: 5 kg, 10 kg, 20 kg, and 30 kg. Knoop indentation loads that yield similar precracks with small lateral crack were chosen. The specimen then was precracked with 2 variations of tilted angles, that are 0.5^o and 1^o in order to observe in which angle would be easier to discern and also to investigate the effect of tilted angle to the value of fracture toughness. After precracking, the residual stress and lateral crack under the indentation impression must be removed. The standard

C1421 introduced the amount removed should be 0.150d-0.167d which is d is length of the long diagonal of Knoop impression and the removing process was done by using hand grinding. The four-point flexure was employed in this research. The loading rate was applied at 1 mm/min. Fracture toughness was computed in accordance with ASTM C 1421. It is also included in this research to investigate the effect surface removed on fracture toughness below and above suggested by the standard.

3. Result and Discussion

The detail of the precracks obtained from different indentation loads is presented in Fig. 1. The suitable precracks were obtained from Hardness Knoop with indentation load 5 kg (HK5) and with load 10 kg (HK10) as can be seen at Fig. 1a and 1b. The precracks were found contain only small size of damage zone and short-shallow lateral crack, therefore only small amount of surface removal needed. In contrary with load 20 kg (HK20) and 30 kg (HK30), the precracks were found irregular, contain large damage zone with long and deep lateral cracks. Some of the precraks in these loads were found with second deep lateral cracks. Since lateral cracks may interfere with the primary median crack and cause errors in determination of fracture toughness [1,4], and with 0.150d - 0.167d will not be sufficient to completely remove the lateral crack, then HK20 and HK30 were not used in this research. Result of precrack size obtained from serial section also useful to facilitate the location and measurement of the precrack size on the fracture surfaces by fitting it in the fracture surface.
Figure 2 shows the effect of fracture toughness, as a function of material removal to be compared with one that obtained from SEVNB method. The fracture toughness of specimens with a surface removal in the range suggested from ASTM C 1421 was found to agree with the results obtained from SEVNB. A surface removal below the recommendation resulted in low values of fracture toughness. Increasing the amount of surface removal moderately was found to still fit with results obtained from SEVNB. Surface removal of much more from the recommended amount leads to failure from natural flaws.
To recognise whether the failure is caused by the precrack or by natural flaws, one can observe the crack propagation after specimen broken. The specimen will broke in to three pieces if failure caused by natural flaws as can be shown in Fig. 2 . It is also possible to be done by observation of fracture surface. If the specimen failure caused by the precrack the fractography on the fracture surface will appear with quite large size fracture mirror.
The influent of several parameters on fracture toughness by using SFC method to be compared with SEVNB method can be observed on Fig. 3. The fracture toughness for SCF method reach the peak value when amount of surface removal approaching the maximum amount surface removal designated by the standard (0.167d). From this peak value, if amount of surface removal was increased, the values of fracture toughness were found decrease. In general, the fracture toughness value obtained with 0.5^o tilted angle resulting higher values comparing with one that have tilted angle 1^o.
It is still found recently that fracture toughness tests for silicon nitride [12,13,14] were conducted by using indentation fracture test method which is not appropriate method since the indentation method is non standardized method for fracture toughness test. It is suggested to use improved SCF standardized method that is introduced in this article

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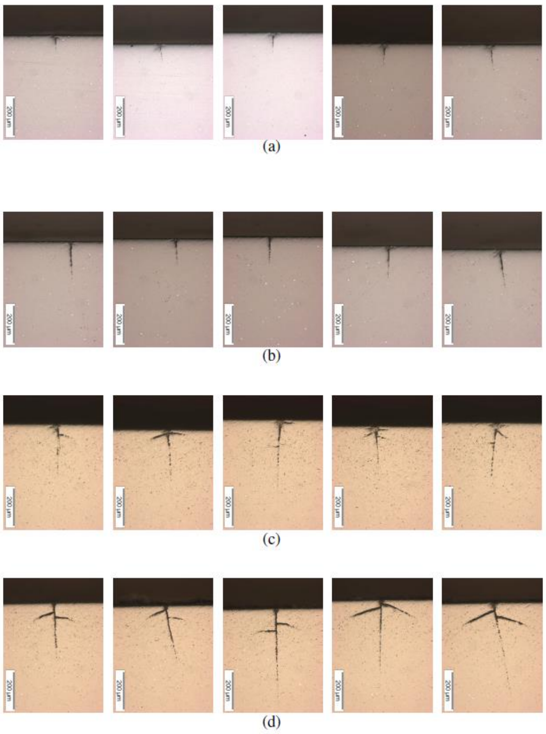

Figure 1. Typical of suitable precracks that contain small size of damage zone with short lateral cracks were obtained from HK5(a) and HK10(b). An irregular form of lateral crack and second deep lateral crack arise from HK 20 (c). A long and deep lateral crack appeared from HK30 (d).

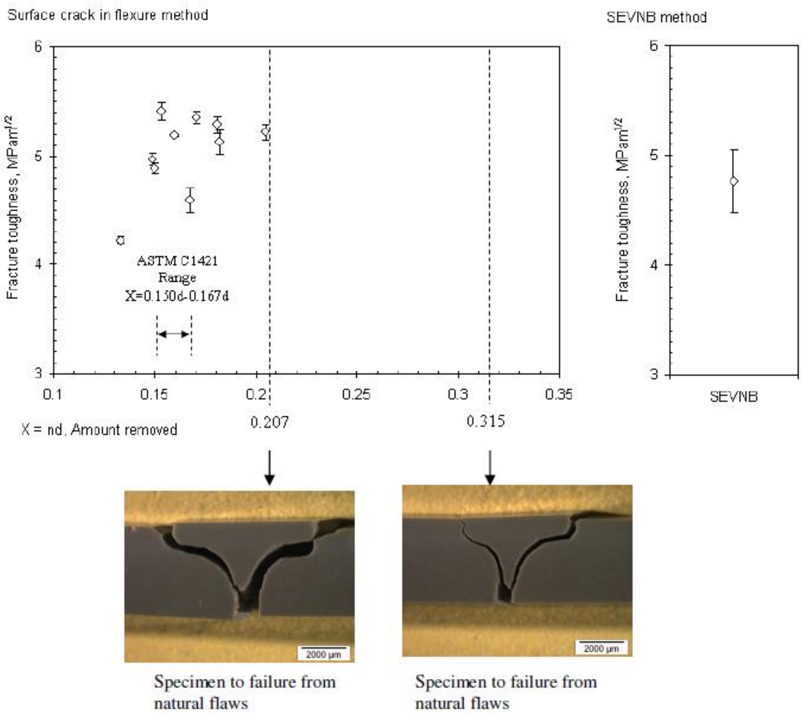

Figure 2. The effect of amount surface removed on fracture toughness in SCF method to be compared with SEVNB methods. Surface removal of much more (0.207d and 0.315d) from the recommended amount leads to failure from natural flaws

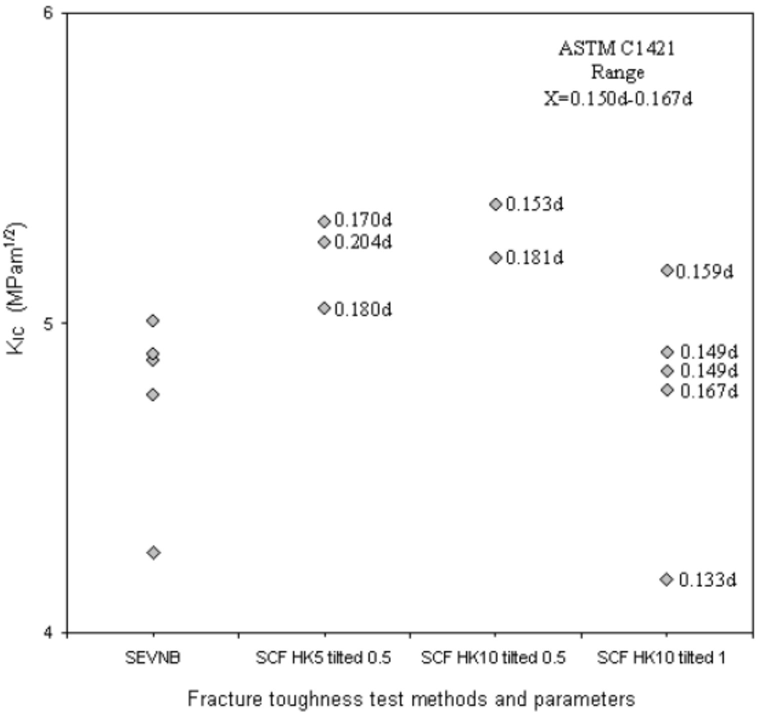

Figure 3. The effect of indentation loads, tilted angles, and amount surface removed on fracture toughness

4. Conclusion

By Serial section is useful to facilitate the location and measurement of the precrack size on the fracture surfaces. Other benefits obtained from serial sectioning is the amount of surface removing also can be determined. From precrack shape that obtained from serial sectioning a suitable indentation load can be selected to avoid irregular or large lateral cracks that may interfere the fracture toughness value.

The fracture toughness of specimens with a surface removal in the range suggested from ASTM C 1421 was found to agree with the results obtained from SEVNB. A surface removal below the recommendation resulted in low values of fracture toughness. Increasing the amount of surface removal moderately is found to still fit with results obtained from SEVNB. Surface removal of much more from the recommended amount leads to failure from natural flaws. The fracture toughness value obtained with 0.5^o tilted angle for the SCF method resulting higher values comparing with one that have tilted angle 1^o.

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