

Fracture Toughness of Silicon Nitride Measured by The Surface Crack in Flexure (SCF) Test Method

(Result Obtained by Using Short Specimen: 3 x 4 x >25 mm)

Tjokorda Gde Tirta Nindhia
Department of Mechanical Engineering
Udayana University,
Jimbaran, Bali, Indonesia, 80361
e-mail:nindhia@yahoo.com

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 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 the amount of material removed from the surface. Short specimens with size 3 x 4 x >25 mm were prepared for this purpose. 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.

Key words: fracture toughness, single edge V-notch beam, surface crack in flexure, silicon nitride.

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. Other publication about investigation the effect of crosshead speed on the fracture toughness of Si_3N_4 reported that: when 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 surface removal 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 Ceramtec (Plochingen, Germany) under the name SL200 B. It is a gas pressure sintered ceramic containing ~ 3 wt.% Al_2O_3 and ~ 3 wt.% Y_2O_3 .

SCF, The bend specimen for this purpose was prepared with cross section size of 3 mm x 4 mm and length of ≥ 25 mm. 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 tilted angle 0.5° order to easier to discern. 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.

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 obtained for valid condition regarding the standard. Fracture toughness was computed in accordance with ISO/CDIS 23146 and the value was compared to the one that obtained from SCF method.

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 precracks 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. If the specimen broken in the valid rule, then the crack propagation will appear as depicted in Fig. 3a for SCF method or as shown in Fig. 3b for SEVNB method. 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 as depicted in Fig. 4a.

The influent of several parameters on fracture toughness by using SFC method to be compared with SEVNB method can be observed on Fig. 5. 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.



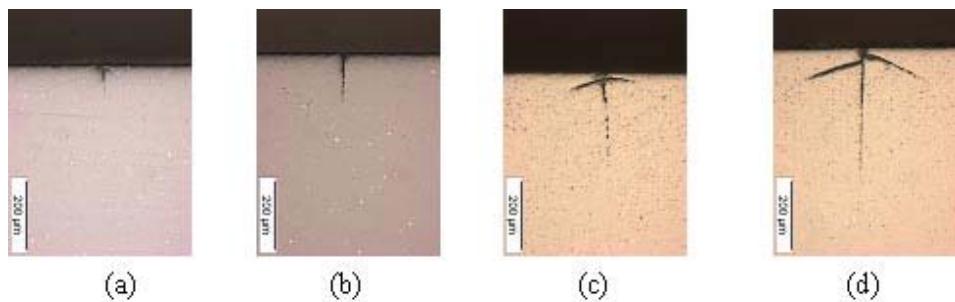


Figure 1. A typical of suitable precracks that contain small size of damage zone with short lateral cracks was obtained from HK5 (a) and HK10 (b). An irregular form of lateral crack and second deep lateral crack arise from HK 20 (c). A long and deep lateral crack appeared from HK30 (d).

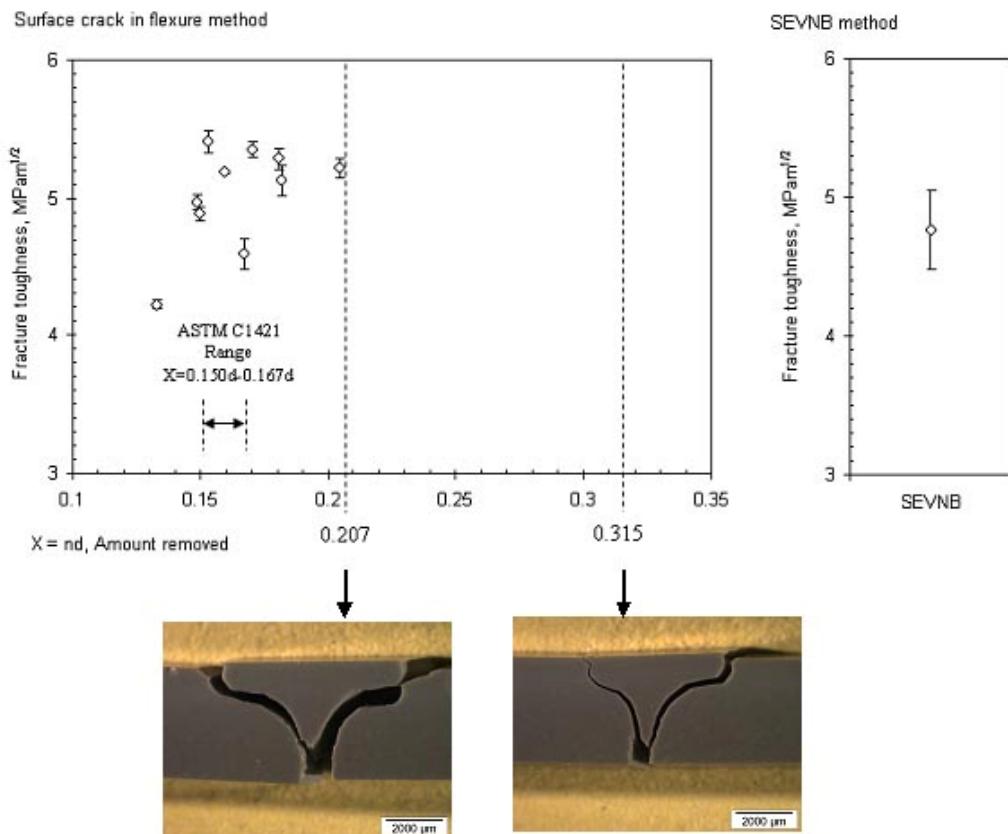


Figure 2. The effect of amount surface removed on fracture toughness in SCF method to be compared with SEVNB methods. Surface removal of much more (0.207d and 0.315d) from the recommended amount leads to failure from natural flaws





Figure 3. Valid crack propagation from SEVNB method (a). Valid crack propagation from SCF method (b)

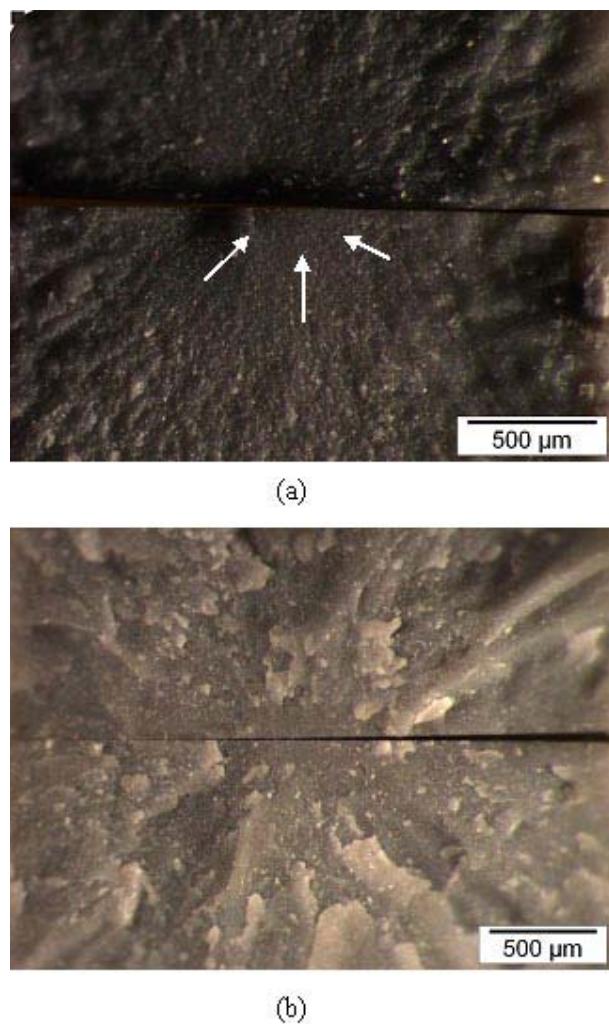


Figure 4. Comparison of fracture surfaces affected by precrack on SCF method (a) Semi elliptical precrack can be found at the centre fracture mirror on SCF method (signed by white arrows) with one that failure from natural flaws (b).



4. Conclusion.

By using short sample with size 3 mm x 4 mm and length of \geq 25 mm, 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.

5. Future work

The hand grinding to remove the residual stresses that was used in this work as recommended by ASTM was indeed time consuming and may take the surface uneven or not parallel to the opposite specimen face. This can cause misalignments during subsequent testing on flexures fixtures. A more efficient procedure was introduced by Quinn and Salem [1] that is by using a flat, circular 30 μm diamond-impregnated abrasive grit plate on a low speed, 200 mm diameter rotary polishing machine, with light finger pressure was introduced. A better method and technique to remove residual stress and lateral crack is in developing as our future work.

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