M4-005 Knoop Indentation Crack Profile in Silicon Nitride

Tjokorda Gde Tirta Nindhia

Department of Mechanical Engineering
Udayana University
Kampus Unund, Jimbaran, Bali, Indonesia
Phone/FAX: 0061-361-703321 ,
E-mail: tirta.nindhia@me.unud.ac.id / nindhia@yahoo.com

ABSTRACT

It is well known that crack obtained from Knoop indentation is often used to introduce initial crack for standardized fracture toughness test of surface crack in flexure (SCF) method for engineering or advanced ceramic. The crack indentation profile obtained is in the form of semi elliptical in shape (or like a section of circle), accompanied with indentation impression, damaged zone, lateral crack and more even chipping is possibly to occur.. The indentation impression, the damaged zone, and the lateral crack need to be eliminated in order a valid elliptical crack to obtain. This can be done by mild grinding, hand grinding, or hand polishing with abrasives papers. The purpose of this work is to reveal the crack profile as a result from Knoop indentation. The result can be applied in investigation of how much the surface at indentation surface should be eliminated in order valid elliptical crack to occur in silicon nitride (Si_3N_4). The research was conducted in four variable load indentations, namely: 5, 10, 20, 30 kg on well polished silicon nitride. Serial sectioning with diamond powder then was done to reveal the crack profile beneath the surface. The crack detail then was plotted. The crack obtained from 5 and 10 kg load of indentation was found without lateral crack with very shallow damaged zone. There fore only small amount should be removed from the surface of indentation (in the range suggested by the standard i.e. 4.5 to 5.0 of indentation deep). It is also revealed that with load of 20 and 30 kg large and irregular lateral crack to occur and second deep lateral crack was found. This is informed that with load of 20 and 30 kg is not appropriate to applied for creating initial crack for SCF fracture toughness test.

Keywords: Knoop Indentation, Crack Profile, Surface Removal, 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 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 was introduced in this research to reveal the crack profile as a result from Knoop indentation. The result can be applied in investigation of how much the surface at indentation surface should be eliminated in order valid elliptical crack to occur in silicon nitride (Si_3N_4).

2. Experimental

The material chosen was silicon nitride produced by Ceram Tec (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.

The serial sectioning was conducted according schematic on Fig. 1, with 4 variations of Knoop indentation loads namely: 5 kg, 10 kg, 20 kg, and 30 kg. The indentations were made on mirror like polished surface. A hardness tester (Zwick 3212) was used for this purpose. During the indentation process the maximum load was set on 5 second with dwelling time was 10 second.

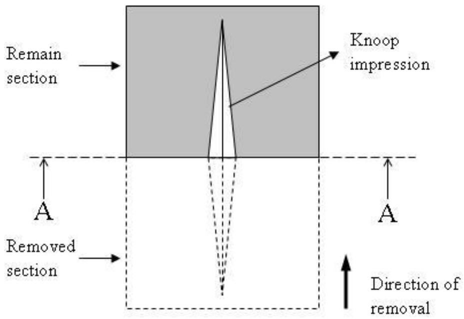


Fig 1. Schematic of serial sectioning process

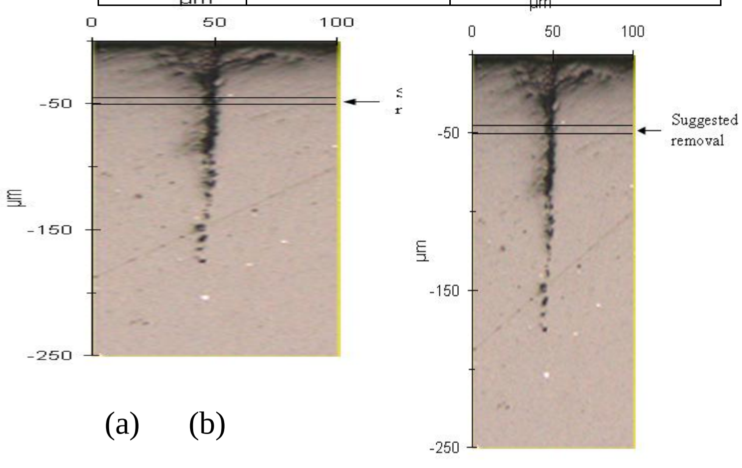
Section-by-section removal was started in a plane perpendicular to the indented surface to the direction of long diagonal. An automatic polishing equipment (Struers Pedemax 2, Denmark) was utilized for the section-by-section removal. For each section two grinding step with resin bonded diamond grinding disc (Struers MD- Piano 220 and 600, Denmark) and 4 polishing step from 15 µm to 1 µm polycrystalline diamonds (Struers DP-suspension P, Denmark) were performed. The crack depths obtained section by section were measured and plotted. The precrak geometries at the middle (section A-A) were analysed.

3. Result and discussion

The detail of the precracks obtained from different indentation loads is presented in Fig. 2-4. 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. 2 and 3. 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 (Fig. 4). 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 sufficient to completely remove the lateral crack, then HK20 and HK30 were not recommended to apply. 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.

Table 1. Calculation of amount of surface removal recommended by ASTM C 1421

Hardness Knoop (HK)	Average length of long diagonal Knoop impression d (µm)	Suggested surface removal according ASTM C 1421 0.150d-0.167d (µm)
5	231.61	34.741-38.602
10	303.20	45.480-50.533
20	466.34	69.952-77.723
30	586.77	88.016-97.795



(a) (b)

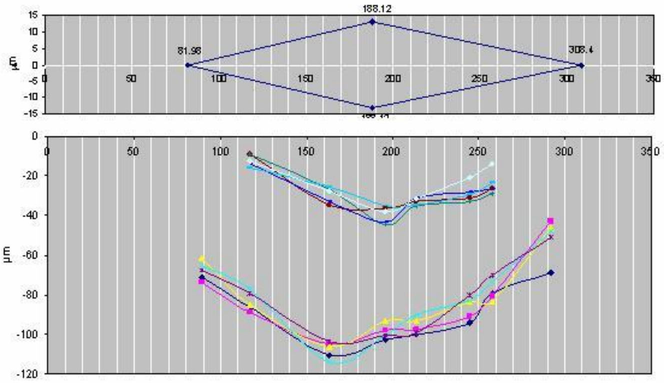


Fig. 2. (a) The precrak geometry at the middle (section A-A of Fig. 1) Typical of suitable precracks that contain small size of damage zone with short lateral cracks were obtained from HK5. (b) Crack depth and damage zone sizes as result from serial sectioning from HK5

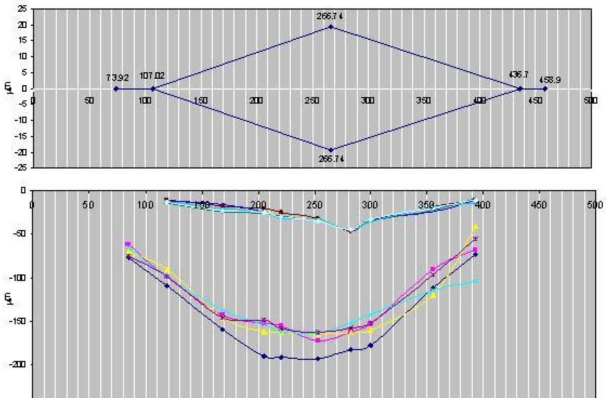
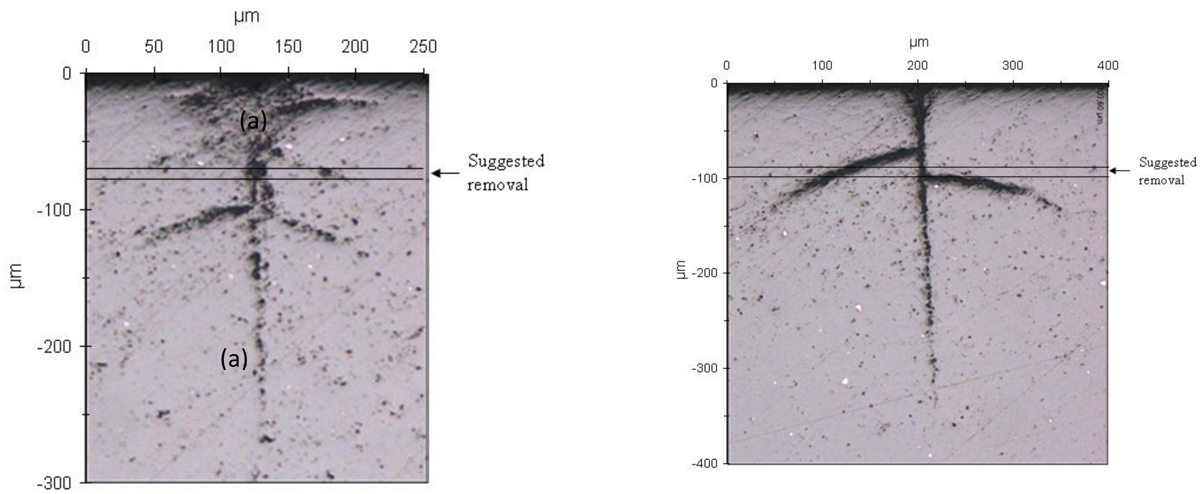


Fig. 3. (a) The precrak geometry at the middle (section A-A of Fig. 1) Typical of suitable precracks that contain small size of damage zone with short lateral cracks were obtained from HK10. (b) Crack depth and damage zone sizes as result from serial sectioning from HK10



(a) (b)

Fig.4. An irregular form of lateral crack and second deep lateral crack arise from HK 20 (a). A long and deep lateral crack yield from HK30 (b).

4. Conclusion.

Serial section is useful to determine the load indentation to be selected for creating precrack and facilitate the location and measurement of the precrack size on the fracture surfaces. Other benefit obtained from serial sectioning is the amount of surface removal 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. For silicon nitride used in this research Knoop indentation of 5 kg (HK5) and 10 kg (HK10) is appropriate to introduce precrack that yield semi elliptical precrack

Acknowledgements

The author acknowledge Prof. Dr. Robert Danzer and Assistant professor Dr. Tanja Lube of *Institut für Struktur und Funktion Keramik, Montanuniversität*, Leoben, Austria on supervising and facilitating this research under scheme of Association of South East Asia-European Union Networking Program..

References

[1] Lube T. Indentation crack profiles in silicon nitride, Journal of The European Ceramic Society 21 (2001)211-218.

[2] Quinn JB, Lloyd IK. Comparison of methods to determine the fracture toughness of three glass-ceramics at elevated temperature, J. Am. Ceram. Soc., 83 [12] 3070–76 (2000).

[3] Quinn GD, Swab JJ, Hill MD. Fracture toughness by the surface crack in flexure (SCF) method: new test results, Ceram. Eng. Sci. Proc., 18 [4] 163–72 (1997).

[4] Quinn GD, Salem JA. Effect of lateral cracks on fracture toughness determined by the surface-crack-in-flexure method, J. Am. Ceram. Soc., 85 [4] 873-80 (2002).

[5] ASTM C1421-99, Standard test method for determination toughness of advanced ceramic at ambient temperature.

[6] Quinn GD, Swab JJ. Fracture toughness of a toughened silicon nitride by ASTM C 1421, J. Am. Ceram Soc. 86[6]1043-45 (2003).

[7] Getting RJ, Quinn GD. Surface crack in flexure (SCF) measurement of the fracture toughness of advanced ceramic, Ceram. Eng. Sci. Proc. 16 [4] 539-47(1995).

[8] Quinn GD, Getting RJ, Kübler JJ. Fractography and surface crack in flexure (SCF) method for evaluating fracture toughness of ceramic, pp.107-44, ceramic transaction, Vol. 64, Fractography of glass and ceramic III, Edited by Varner JP, Frech VD, Quinn GD, American Ceramic Society, Westerville, OH, 1996.

[9] Park JK, Yasuda K, Matsuo Y, Effect of crosshead speed on the fracture toughness of soda lime glass, Al_2O_3, and Si_3N_4 ceramics determined by the surface crack in flexure (SCF) method. Journal of Material Science, 36 (2001) 2335-2342.