FRACTURE TOUGHNESS OF SILICON NITRIDE EVALUATED BY IF, IS AND SEVNB METHODS

J. Špaková, J. Dusza

Abstract
The aim of the present study is to compare fracture toughness values obtained using two indentation methods with the conventional fracture toughness testing methods. Fracture toughness of the monolithic Si_3N_4 was evaluated by indentation fracture toughness (IF), indentation strength (IS) and single-edge-V-notched-beam test (SEVNB). The value of the fracture toughness by SEVNB was 5.12 ± 0.39 $MPa \cdot m^{1/2}$. In the case of indentation methods, the fracture toughness has been measured at loads from 49.05 N to 196.2 N. IS toughness values were in the range 4.71 ± 0.18 to 5.19 ± 0.33 $MPa \cdot m^{1/2}$, which is in good agreement with SEVNB values. IF method led to consistently higher values compared to SEVNB and IS methods: from 6.11 ± 0.66 to 6.5 ± 0.23 $MPa.m^{1/2}$.
Keywords: Si_3N_4, fracture toughness, indentation fracture toughness, indentation strength, single-edge-V-notched-beam technique

INTRODUCTION

Silicon nitride based ceramics are considered as promising structural materials in different fields of industry because of their high hardness, wear resistance and strength, good oxidation and excellent thermal shock resistance. Using microstructure design (self-reinforcement by large elongated grains and tailoring the chemistry of the intergranular phase) a room temperature fracture toughness of more than 4 $MPa \cdot m^{1/2}$ and fracture strength higher than 800 MPa have been achieved [1]. One of the successful applications of the developed Si_3N_4 are tribological components in corrosive surroundings, such as bearings for hard disks and mechanical seals, etc. The assessment of the fracture toughness of such components is among the most important parameters for their design and reliability. There are several methods to determine fracture toughness of brittle materials including glasses, glass ceramics and advanced ceramics. These involve indentation techniques (indentation fracture method **"**IF**"** [2], indentation strength "IS" [3], surface cracks in flexure **"**SCF**"** [4]), chevron notch beam technique "CNB" [5], single edge V-notch beam technique "SEVNB" [6], single edge precracked beam technique "SEVPB" [7], etc**.** The purpose of the present work is to compare three fracture toughness evaluation methods, indentation fracture, indentation strength "IS" and single edge V-notch beam technique "SEVNB" in order to determine reliability of indentation techniques. Both indentation methods to determine fracture toughness, IF and IS, are based on the empirical calibration constants; hence they are less rigorous theoretically from a fracture mechanics perspective than the other methods. IF method is currently used to measure fracture toughness based on length measurements of cracks introduced by the Vickers indenter. The main advantage of IF method is that it is very easy to perform and it requires small samples. On the other hand, it is less reliable and the final result depends on many factors, such as the surface roughness, the way of the crack size measurement, and sensitivity to postindentation cracks

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extension. Furthermore, depending on the crack shape, many equations for calculation of the fracture toughness [8] exist. The result depends on the equation chosen for the calculation of the fracture toughness. The indentation strength method “IS” is a two step technique that requires the introduction of a crack by hardness indentation and controlled fracture in bending. Single - Edge V-Notched Beam Technique (SEVNB) is a standardized measurement technique.

MATERIALS AND EXPERIMENTAL METHODS

The monolithic Si_3N_4 (SL20-B, CeramTech Plochingen, Germany) was gas pressure sintered with addition of 3 wt.% Y_2O_3 and 3 wt.% Al_2O_3.

The tensile surface of each specimen was polished to a mirror finish using diamond paste. The samples for the four-point bending in the IS and SEVNB tests were 50 mm long, 4 mm wide and 3 mm thick. The edges of the tensile surface of the specimens were slightly chamfered by abrasive papers.

Indentation fracture toughness method (IF)

This method includes measurement of the length of generated cracks on the specimen surface after Vickers indentation until acceptable crack patterns are obtained. The criteria for acceptability are: all crack generated from the corner of the Vickers indent, presence of only four radial cracks, no crack chipping and no branching. The values of indentation fracture toughness have been measured in wide range of applied loads from 50 N to 200 N and dwell time of 10 seconds on device HPO 250 hardness tester with Vickers indenter. The average values for indentation fracture toughness were calculated from 10 indents. Two different experimental techniques were used to investigate the cracks patterns: serial sectioning technique [9] and decorating process [10].The values of the fracture toughness were calculated according to the most frequent equations (Table1).

Tab.1. The most frequent equations for calculation of K_{IC} from Vickers indentation cracks system.

Autor and ref.	Shape of crack	Equation for K_{IC}
Antsis et al. [11]	Half-penny	$0,016.\left(\frac{E}{H}\right)^{\frac{1}{2}}.\frac{P}{c^{\frac{3}{2}}}$
Shetty [12]	Palmquist	$0,0889.\left(\frac{H.P}{4l}\right)^{\frac{1}{2}}$

H – Vickers hardness [GPa] c – length of the cracks [m]
P – applied indentation load [N] d - length of the indent`s diagonal [m]
E – Young`s modulus [GPa] l = c-d

Indentation strength method (IS)

Vickers indentation was located in the centre of the tensile surface of each specimen using the load in the range 100 N - 200 N. Five specimens were indented at each load. Indented samples were subjected to a four point bend test (Loyd LR5K PLUS). Fracture toughness was calculated from a unified indentation strength equation:

$$K_{IC} = \eta.\left(\frac{E}{H}\right)^{1/8}.\left(\sigma_f.P_i^{1/3}\right)^{3/4} \quad . \tag{1}$$

η= 0.59 σ_f – stress at fracture
E -Young`s modulus P – applied indentation load
H – hardness

Single - Edge V-Notched Beam Technique (SEVNB)

V-notches were machined into five specimens. The cut was introduced by a diamond saw and this notch was tapered by a razor blade with 3 μm diamond paste. Thus a sharp notch with tip radius of 10 μm was produced. Fracture toughness was determined from the residual bending strength of the bars with the V-notch according the following relationships:

$$K_{IC} = \frac{F_{MAX}}{B.W^{1/2}}.\frac{L_1 - L_2}{W}.\frac{3.\alpha.M}{2.(1-\alpha)^3} \tag{2}$$

$$M = 1{,}9887 - 1{,}326.\alpha - \frac{\left(3{,}49 - 0{,}68.\alpha + 1{,}35.\alpha^2\right).\alpha.(1-\alpha)}{(1+\alpha)^2} \tag{3}$$

$$\alpha = \frac{a}{W} \tag{4}$$

σ - 4-point bending strength L_1- outer span distance
F – fracture load L_2- inner span distance
B – specimen thickness a – V-notch length
W – specimen width

RESULTS AND DISCUSSION

In the case of IF method, Fig.1a shows the profile of Palmquist/half-penny cracks (HV 50) revealed by serial sectioning and Fig.1b illustrates half-penny cracks on a fracture surface (HV 100).

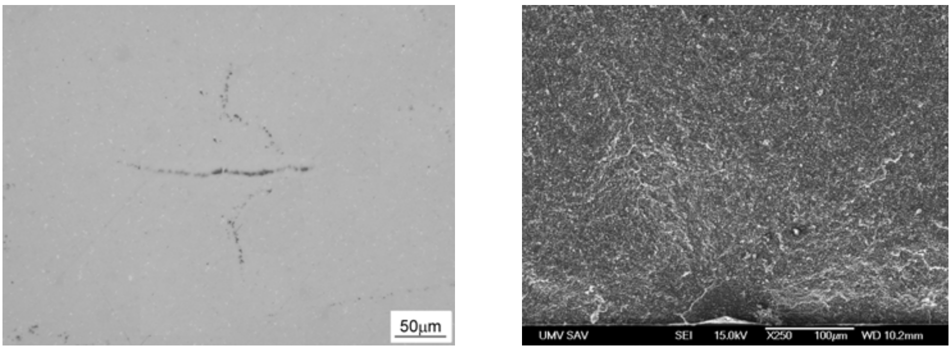

Fig.1. The change of crack`s shape with increased indentation load a) mixed shape (Palmquist/half-penny) at 50 N b) half-penny shape at 100 N.

These figures demonstrate the dependence of the crack shape on the indentation load (at 50 N-mixed: Palmquist/half-penny shape of indentation cracks, at 100 N only half-penny shape). A similar result was reported by Lube [9]. Fracture toughness was calculated according to the equations in Table 1 depending on the type of the cracks. Figure 2 compares K_{IC} values obtained by IF, IS and SEVNB methods. The IF method overestimates K_{IC} when compared to the values measured by the SEVNB method. The discrepancy may be attributed to many factors: determination of the exact crack length, existence of multiple cracks, choice of indentation load and equation, possibility of the postindentation crack extension, etc. Moreover the stress state, geometry, loading conditions and cracks are well defined for the standard SEVNB test and a closed form solution of the stress state is available.

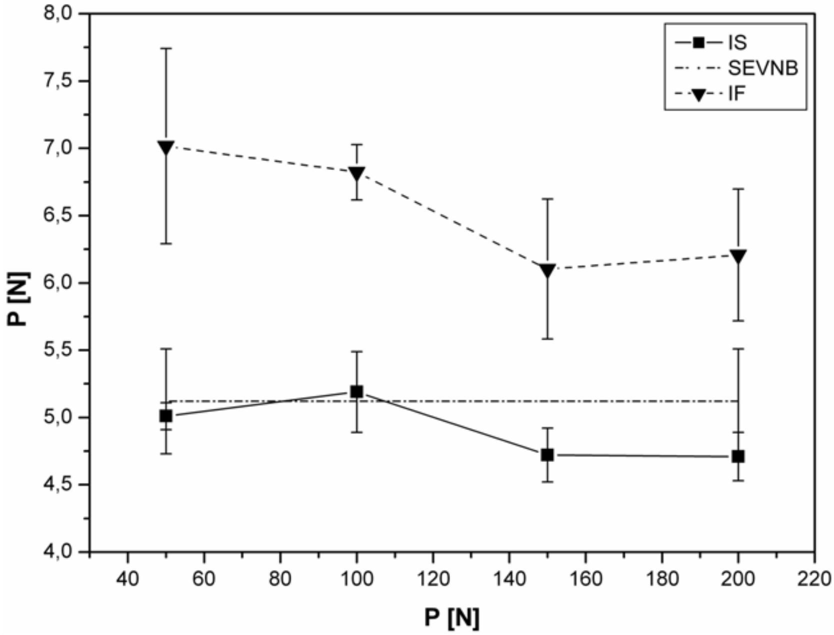

Fig.2. Comparison of K_{IC} indentation techniques vs. standard SEVNB method.

In the case of IF method, any size and shape of specimen are adequate, so long as it can be mounted and polished; there is no single pre-crack in the IF specimen and multiple cracks are generated during loading, the cracks decelerate to an arrest condition away from the indentation. Moreover, the IF cracks do not have an applicable stress intensity solution [13]. After a SEVNB test, the single crack has halved the specimen, but in IF test, multiple cracks are arrested and residual stresses of considerable complexity exist in a post-test configuration. The multiple cracks in a IF test do not remotely ascribe to the definition of fracture toughness [14]. The values of K_{IC} measured by IS method were comparable with the values obtained by the standard method. In this case there is no need to determine the initial crack length, the crack will extend stably during the fracture test in response to the external load and residual stress fields associated with indentation until it reaches a critical size leading to catastrophic failure.

CONCLUSIONS

The IF technique is not a reliable fracture toughness test method for ceramic materials. In contrast an overall agreement between IS and SEVNB methods was obtained. A specific residual stress intensity factor term in the strength/toughness formulation overcomes the systematic error inherent in the IF method, but as it does not adequately account for residual stresses fields, the values of fracture toughness estimated by IS technique are more reliable.

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