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# Effect of Notch-Root Radius on the Fracture Toughness of Composite $Si_3N_4$ Ceramics

*Cláudio Vasconcelos Rocha and Célio Albano da Costa*

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**In-situ silicon nitride and a whisker-reinforced silicon nitride-silicon nitride composite, densified via gas pressure sintering and hot pressing, respectively, were evaluated using the single-edge V-notched beam (SEVNB) fracture toughness technique. The mean value of $K_{IC}$ for each material was 5.7 and 7.9 MPa · $m^{1/2}$, respectively, and the toughness was influenced by the presence of the elongated $Si_3N_4$ grains in the microstructure. The notch radius was observed to have the same effect as a sharp crack when notch-root radius was smaller than 10 μm, which was considered to be a real $K_{IC}$ for these materials.**

**Keywords** fracture toughness, gas pressure sintering, hot pressing, notch-root, silicon nitride

## 1. Introduction

Fracture toughness ($K_{IC}$) is a very important mechanical property, and it has been extensively used to characterize advanced ceramics. The major difficulty in the measurement of $K_{IC}$ in ceramics is to simulate a "real" sharp crack, which can subsequently be opened by a reliable and easy procedure (Ref 1). Different from metals, where fatigue is used to initiate and grow a sharp crack in a controlled manner, in ceramics such a method is not feasible (Ref 2).

The methods used to measure $K_{IC}$ in ceramics can be divided in two groups: those that try to introduce a real crack and those that simulate a sharp crack (Ref 3). For instance, surface crack in flexure (SCF) or single-edge precracked beam (SEPB) belong to the first group, and chevron notch (CN) and single-edge notch beam (SENB) to the second. The first set of techniques is characterized by the difficult nature of opening the crack and measuring it after the test, which was considered to be almost impossible by Quinn et al. (Ref 4). For the second group, the problem lies in obtaining a notch with a root-radius similar to a real crack.

A recent technique that has shown very good results is the single-edge V-notched beam method (SEVNB) (Ref 5). This method consists of introducing a notch with a thin diamond wheel and, subsequently, polishing it with a razor blade, which is embedded in diamond paste, until the notch-root forms a V with a very sharp root radius (Ref 6). The SEVNB is a fracture mechanics test that can lead to a simple and reliable method of measuring $K_{IC}$.

The main objective of this study was to characterize two types of composite silicon nitride by SEVNB. The materials were densified either by gas pressure sintering (GPS) or hot-pressing (HP) and resulted in microstructures with different grain size, aspect ratio, and, consequently, fracture toughness. The effect of the notch-root radius on the fracture toughness of both materials was also examined.

**Cláudio Vasconcelos Rocha** and **Célio Albano da Costa,** Programa de Engenharia Metalúrgica e de Materiais (PEMM/COPPE), Centro de Tecnologia, Bloco F, Sala 210, Universidade Federal do Rio de Janeiro, Rio de Janeiro, RJ, 21949-900, Brazil. Contact e-mail: celio@metalmat.ufrj.br.

## 2. Experimental Procedures

Silicon nitride ($Si_3N_4$) was homogenized with two types of additives by colloidal processing (i.e., ball milled with $Si_3N_4$ balls in alcohol for 12 h, dried, and then sieved), uniaxially pressed at 30 MPa, and then densified using either GPS or HP. The GPS material, composed of $\alpha$-$Si_3N_4$, 5% $Y_2O_3$, and 3.5% $Al_2O_3$, was sintered at 1850 °C for 30 min under an $N_2$ pressure of 10 MPa. The HP material was composed of $\alpha$-$Si_3N_4$, 10% $\beta$-$Si_3N_4$ whiskers, and 3% MgO and was hot-pressed at 1750 °C for 2 h at 32 MPa under a $N_2$ atmosphere.

After sintering, the specimens were cut and surface ground to 3 × 4 × 50 mm (W × B × L), as recommended in ASTM C-1161 (Ref 7). Preparation of SEVNB specimens consisted of opening a straight notch centered on the 3 mm width surface to a depth 0.5 mm using a 0.15 mm thick diamond wheel. The next step increased the depth of the notch by the reciprocating movement of the razor blade sprinkled with diamond paste (1 μm). This procedure resulted in a V-shaped notch where the root radius is expected to have the same effect as a sharp crack, which then allows reliable determination of $K_{IC}$. The sharp V notches were introduced on a plane jig supported by springs (Fig. 1), which meets the following requirements: the fixture must have high stiffness to prevent any twisting or bending of the blade as it passes over the specimens and maintains a consistent orientation during the reciprocating movement of the machine. Use of the springs in the jig allowed the adjustment of the normal force applied on the specimen because the machine table controls the depth of the razor blade. To control the notch-root radius and its depth, all specimens were subjected to the following procedure before the flexure test: (1) clean in ultrasonic bath with acetone to remove particle/clusters of the diamond paste, which could be ingrained at the notch root, and thus, impair the precise measurement of the notch radius; and (2) place the specimens in the scanning electron microscopy (SEM) and measure the notch-root radius and depth. It is worth mentioning that both sides of each specimen were measured.

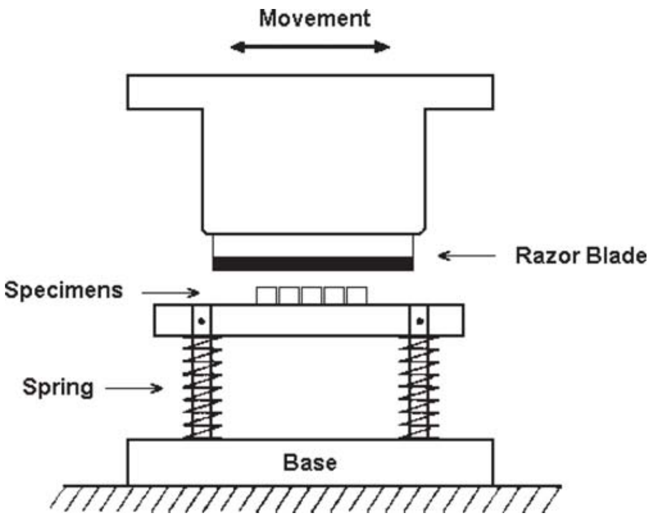


**Fig. 1** Schematic diagram of the assemblage used for opening a sharp notch with the razor-blade polishing technique

The specimens selected for fracture toughness testing were those that had a notch-root radius and depth difference on both sides smaller than 10 μm and 0.2 mm, respectively. Tests were then conducted in four-point bending with a cross-head speed of 0.05 mm/min. The deflection of the specimens was measured using an LVDT.

To observe the grain structure, the surfaces of the specimens were polished and then etched with HF at 112 °C. These resulting microstructures were observed in the SEM and digitally analyzed using IntelligentView, which identifies the grain boundary pixels and then measures the grain length and thickness of each grain. The quality of the grain size measurement is directly related to the clear identification of the boundary, and many convolution steps were necessary to achieve consistent results. About 560 and 1200 grains were identified for the GPS and HP materials, respectively.

## 3. Results and Discussion

The fracture toughness of $Si_3N_4$ is affected significantly by the microstructure. For instance, the presence of elongated grains in a fine equiaxed matrix results in high fracture toughness because crack deflection and crack bridging are dominant toughening mechanisms (Ref 8). Also, the characteristic microstructure of the "in situ" reinforced composites gives a high Weibull modulus (Ref 9), a desired parameter in mechanical design. Because the grain size is an important microstructural parameter, its quantification and effect on $K_{IC}$ are very much desired.

The microstructural quantification of the HP material showed the grain length and the aspect ratio to be 23% higher than the GPS (Table 1), while the grain width is pretty much the same for both. When grain length distribution (Fig. 2) was taken into account, it was observed to be unimodal for both materials. However, the HP sample had a much broader distribution (0.2-5 μm) with a large fraction of grains longer than 2 μm. The GPS sample had a narrow distribution (0.1-3.7 μm), with a peak centered at 0.6 μm. The longer HP grains are a direct consequence of the 10 wt.% whiskers, which were intentionally added to increase the fracture toughness, while in the GPS sample, the grains were formed "in situ" by the $\alpha \rightarrow \beta$ transformation.

**Table 1 Microstructural features of the analyzed samples**

| Sample | Length, μm | Width, μm | Aspect ratio |
|---|---|---|---|
| GPS | 1.07 | 0.50 | 2.16 |
| HP | 1.32 | 0.52 | 2.67 |
| HP/GPS | 1.23 | 1.04 | 1.24 |

The fracture toughness of ceramic materials is very sensitive to the notch root radius. For instance, Damani et al. (Ref 2) and Nishida et al. (Ref 10) showed that for dense polycrystalline ceramics with a small grain size, such as silicon nitride, a notch-root radius ($\rho$) smaller than 10 μm can simulate a sharp crack and allows a reliable determination of $K_{IC}$. In the case of higher $\rho$, the fracture toughness increases linearly with $\rho^{1/2}$ (Ref 3). Kübler (Ref 11) empirically proved that SEVNB fracture toughness values can be considered true when the curvature of notch-root radius is smaller than twice the size of the largest microstructural feature of the material, mostly represented by the grain size ($d$). To contribute to a better understanding of this phenomenon, five samples of the GPS and HP materials were tested using the SEVNB method, as shown in Fig. 3. For illustration purposes, the largest and smallest notch-root radius of each material is shown in Fig. 4.

The average $K_{IC}$ of the GPS samples was 5.69 MPa · $m^{1/2}$ with a standard deviation of 0.18 MPa · $m^{1/2}$, while the HP samples were 7.89 MPa · $m^{1/2}$ with standard deviation of 0.08 MPa · $m^{1/2}$. It was clearly observed that the fracture toughness of the HP material is 27% higher than the GPS and this difference can be attributed to the microstructure, because the grain length and the aspect ratio are 23% higher in the HP material. Also, the low value of standard deviation strongly indicates that the SEVNB technique is quite reliable. Similar results were also obtained by Peillon (Ref 8), Kleebe (Ref 14), and Ziegler (Ref 15), whom tested $Si_3N_4$ and observed that the higher values of fracture toughness was related to the high grain length and aspect ratio, confirming the importance of these microstructure features. The higher value of the HP samples was obtained with the notch placed parallel to the hot-pressed direction, where the whiskers act most effectively to toughen the material. If the $K_{IC}$ were measured on a plane perpendicular to the HP direction, where the whiskers in one direction are aligned with crack path, the value would be lower, as reported in the literature (Ref 16-18). For instance, Costa (Ref 19) measured $K_{IC}$ (by the indentation technique) on a $Si_3N_4$ whisker-reinforced $Si_3N_4$ composite and found a difference of about 15% between the direction perpendicular (8.8 MPa · $m^{1/2}$) and parallel (7.6 MPa · $m^{1/2}$) to the HP direction. This demonstrates the anisotropy behavior in HP $Si_3N_4$, in contrast with the isotropy of the GPS material.

The fracture toughness of the GPS material measured by SEVNB (5.69 MPa · $m^{1/2}$) was pretty much the same as other GPS silicon nitrides, as shown in Fig. 3. This is the result of the similar type and amount of sintering aids, the isotropy of the sintering process, and equivalent sintering schedule. On the other hand, the fracture toughness of HP $Si_3N_4$ reported in the literature (5.4 [Ref 2] and 6.0 MPa · $m^{1/2}$ [Ref 20]) was lower

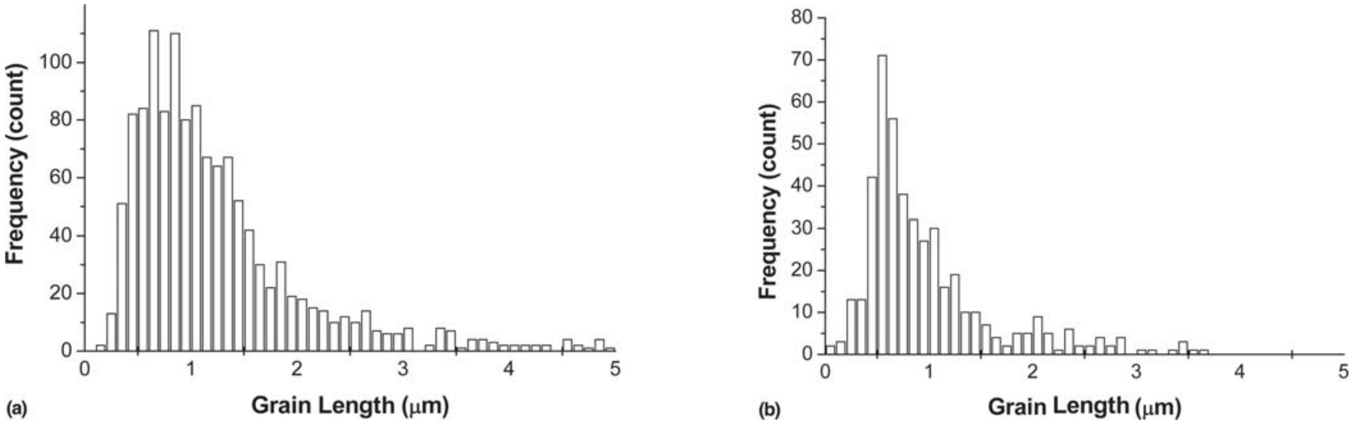


**Fig. 2** Grain length distribution: (a) HP and (b) GPS materials

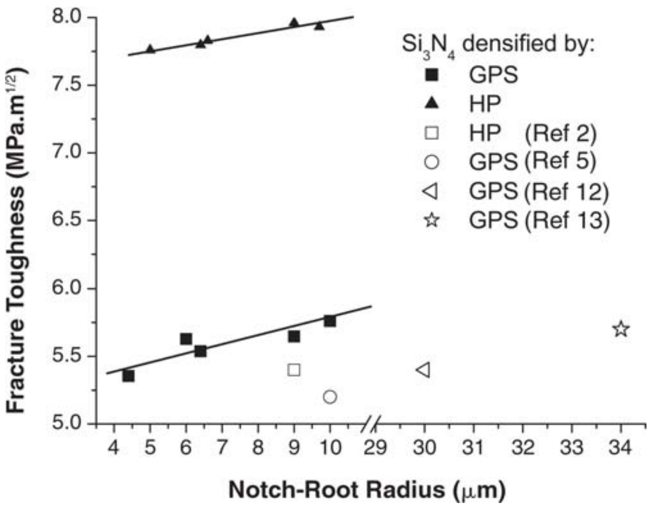


**Fig. 3** Fracture toughness of the GPS and HP materials as a function of the notch-root radius. Open symbols were obtained from literature, correlating the notch-root radius and $K_{IC}$.

than the one measured here (7.89 $MPa \cdot m^{1/2}$). In this case, the high $K_{IC}$ of the present material is the result of added whiskers.

When the grain size of the HP (5 μm) and the GPS (4 μm) materials are compared with the above criterion proposed by Kübler (Ref 11), it was expected that ρ would be smaller than 10 and 8 μm, respectively, to obtain a reliable value of $K_{IC}$. This criterion was followed by all samples, except for one of the GPS group, which was 20% higher. The strict application of the criterion has to be carefully observed, because Kübler himself, testing a GPS $Si_3N_4$, stated that the notch root with a curvature radius smaller than 30 μm produced results for $K_{IC}$ that differed by only 0.56% from those that obeyed the $\rho \leq 2d$ criterion (Ref 11). If the Damani (Ref 2) and Nishida (Ref 10) criteria are used, then all data can be considered valid, because the higher ρ measured was 10 μm. For the time being, a radius of curvature smaller than about 10 μm seems to simulate a sharp crack and provides reliable $K_{IC}$ data. Also, a unique and definitive criterion relating ρ with any microstructural feature is still to emerge.

Closer examination of the $K_{IC}$ versus ρ curve (Fig. 3) shows that as the radius of curvature is reduced from 10 to 4 μm, there is a very small reduction in the $K_{IC}$ value. For the GPS material, the $\Delta K_{IC}$ between the highest and lowest values was 7%, while for the HP material it was even lower (~2%). These differences are small, highlighting the fact that the fracture toughness measured is close to a real and constant value, and shows how strong the influence of ρ is on $K_{IC}$. A similar behavior was noted by Gogotsi (Ref 5) also working with HP $Si_3N_4$ and GPS $Si_3N_4$. He reported that the $K_{IC}$ versus ρ curve asymptotically approached a finite value as the radius of curvature (ρ) became smaller than 10 μm, and only when ρ was lower than 5 μm were constant values of fracture toughness measured. On the other hand, Kübler (Ref 12) found that constant $K_{IC}$ data were obtained when ρ approached 30 μm, almost a twofold increase. When the present data and that from the work of Damani (Ref 2), Gogotsi (Ref 5), Nishida (Ref 10), and Kübler (Ref 11) are consolidated (Fig. 3), it was noted that the relationship between $K_{IC}$ and ρ are congruent, namely, as ρ becomes smaller that 10 μm the $\Delta K_{IC}$ asymptotically approaches a constant value, which can be assumed as a material property.

About the technique itself, it was verified that larger notch-root radius occurred when the razor blade was not periodically changed. The friction of the razor blade with the specimen increased the razor blade thickness and allowed the formation of secondary cracks ahead of the main notch tip. Under externally applied load, such secondary cracks are likely to grow along with the main crack, and as a result, create a dispersion of $K_{IC}$ values. In the present work, the razor blade was changed regularly (every 2 h) with the objective of keeping ρ small; otherwise, a high $K_{IC}$ would be measured, as observed by Mukhopadhyay et al. (Ref 21).

It was observed that the SEVNB technique needs a standard experimental procedure, for instance: (a) The V-shaped notch simulates a sharp crack only if the razor blade is kept sharp during the whole opening process. Thus, a constant change of the razor blade is needed at periodic intervals. (b) Attention should be paid to the crack initiation region to prevent the formation of secondary cracks ahead of the main notch tip, as this can alter the measured $K_{IC}$. (c) Stable crack extension can occur, and if it is not identified, erroneous results will be obtained. If these basic steps are followed, the SEVNB technique can be considered reliable and result in low dispersion of the data. Furthermore, a representative value of ρ is affected by other parameters, a major one being the grain size, the full effect still has not been confirmed.

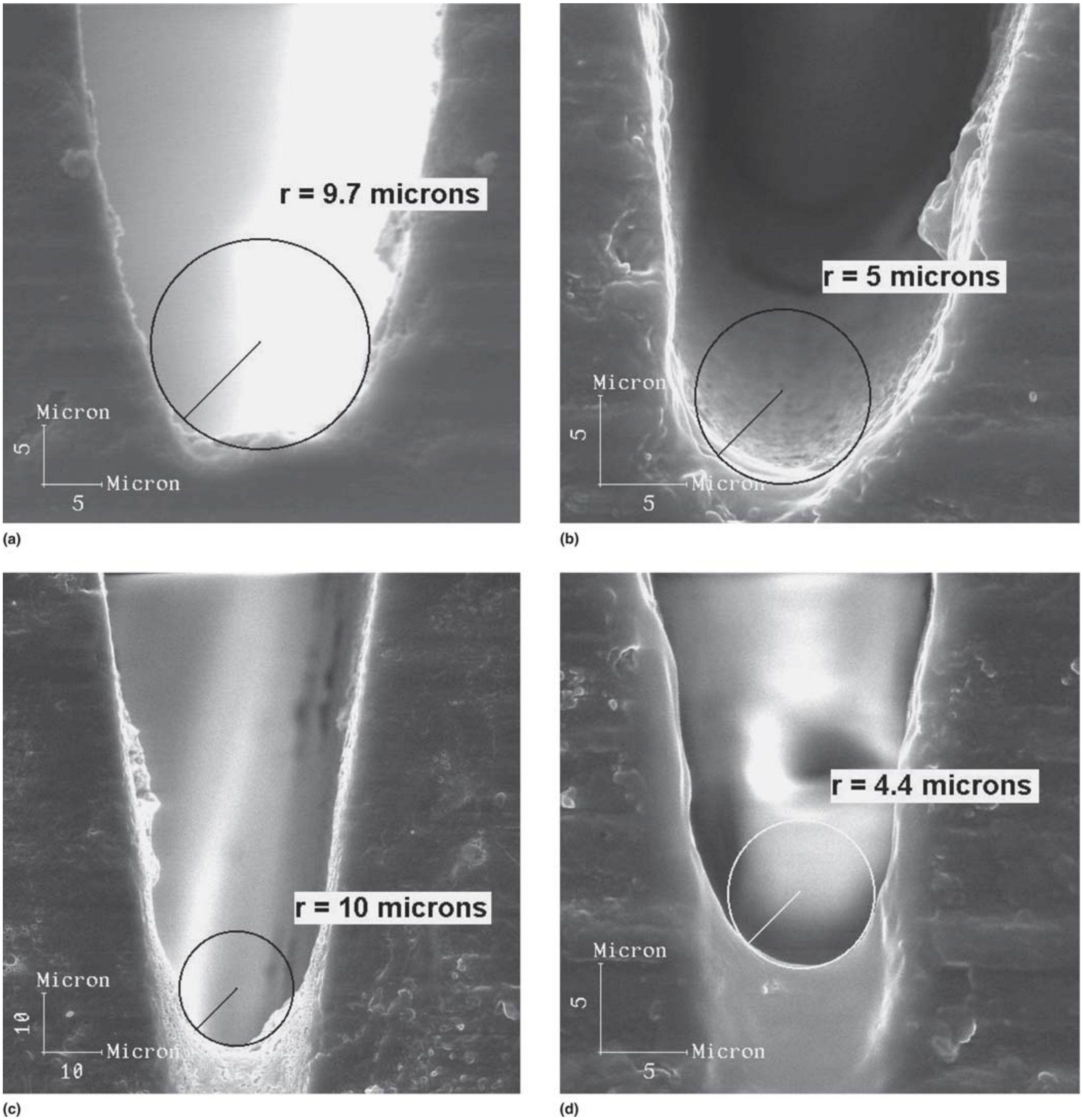


**Fig. 4** SEM of SEVNB notch-root radius: (a) largest and (b) smallest ρ of HP material, respectively; (c) largest and (d) smallest ρ of GPS material, respectively

## 4. Conclusions

It was shown that the fracture toughness of HP material was 27% higher than the GPS, which can be explained by the longer grains and higher aspect ratio. The $K_{IC}$ values can be considered as representative of each material because the coefficient of $K_{IC}$ versus ρ is very small and ρ was lower than 10 μm, which also fits the criterion of $\rho \leq 2d$ (grain size). However, more research still needs to be done to confirm that grain size is the unique microstructure feature that determines the value of ρ. For the time being, a value of ρ below 10 μm seems to be reasonable in the determination of fracture toughness of silicon nitride.

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