FRACTURE TOUGHNESS IN ADVANCED MONOLITHIC CERAMICS -SEPB VERSUS SEVNB METHODS-

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ABSTRACT

Fracture toughness of a total of 13 advanced monolithic ceramics including silicon nitrides, silicon carbide, aluminas, and glass ceramic was determined at ambient temperature by using both single edge precracked beam (SEPB) and single edge v-notched beam (SEVNB) methods. Relatively good agreement in fracture toughness between the two methods was observed for advanced ceramics with flat R-curves; whereas, poor agreement in fracture toughness was seen for materials with rising R-curves. The discrepancy in fracture toughness between the two methods was due to stable crack growth with crack closure forces acting in the wake region of cracks even in SEVNB test specimens. The effect of discrepancy in fracture toughness was analyzed in terms of microstructural feature (grain size and shape), toughening exponent, and stable crack growth determined using back-face strain gaging.

1. INTRODUCTION

There are several methods to determine fracture toughness of brittle materials such as glasses, glass ceramics, and advanced monolithic ceramics. These includes indentation techniques such as indentation fracture (IF) [1], indentation strength (IS) [2], surface crack in flexure (SCF) [3], chevron notch technique [3], single edge precracked beam (SEPB) technique [3], and single edge v-notched beam (SEVNB) technique [4] technique. Both indentation fracture (IF) and indentation strength (IS) methods are based on the empirical calibration constants to determine fracture toughness; hence, they are less rigorous theoretically from a perspective of fracture mechanics than the other methods.

The SEVNB technique [4] has been introduced recently, in which a final sharp v-notch with its radius ranging from 10 to 20 μm was introduced by polishing a pre-notched section with razor blade in conjunction with diamond paste. This technique has shown to be in good agreement with other techniques. A previous round robin on fracture toughness [4] was dedicated to use the SEVNB method to determine fracture toughness of five different ceramic materials. It was shown that certain ceramics exhibited significant difference in fracture toughness between the SEVNB and SEPB methods: the SEVNB estimated lower fracture toughness than the SEPB. The plausible reason for this discrepancy was presumed to be attributed to R-curve behavior of the materials.

This study, as motivated from the previous round robin, determined fracture toughness of a total of 13 advanced ceramics using both SEVNB and SEPB methods. The difference in fracture toughness between the two methods was carefully analyzed based on R-curve estimations and back-face strain measurements. The effect of major microstructural feature - grain size and its shape - was also discussed.

2. EXPERIMENTAL TECHNIQUES

Flexure test specimens typically measuring b=3.0 mm, W=4.0mm, and L (length)=25 or 50 mm, were used in fracture toughness testing at room temperature in air. In the SEVNB method, a razor blade with diamond paste with grain size of 1 μm was used to introduce a final sharp notch with a

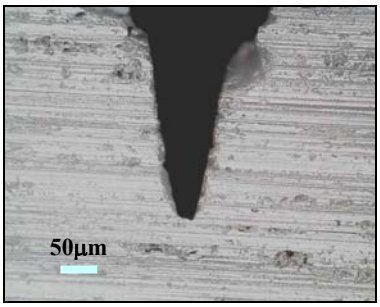

Figure 1. A typical example of a sharp v-notch produced in SEVNB test specimen.

root radius ranging from 10 to 20 μm by tapering a saw notch [4]. The final notch depth thus produced ranged from 1.0 to 2.0 mm. A typical example of a SEVNB test specimen thus v-notched is presented in Figure 1. In the SEPB method [3], a starting indent crack was placed at the center of the 3.0 mm side of each test specimen. The indented test specimen was then placed onto a specially designed precracking fixture and then loaded via a test frame until the indent crack popped-in to form a sharp through-the-thickness crack [3,5]. The precrack size was typically around 2 mm.

A four-point flexure fixture with 20 (or 10) mm-inner and 40 (or 20) mm-outer spans was used to determine fracture load. A test rate of 0.5 mm/min was used via an electromechanical test frame (Model 8562, Instron) with a load cell with a capacity of 1000 N. Crack sizes were optically determined from fracture surfaces of tested specimens. Typically, five test specimens were used for each material in each test method. Fracture toughness was calculated based on the formula by Srawley and Gross [6]. Load vs. back-face strain curves were also determined for some ceramics to better understand stable crack propagation. R-curve of each material was estimated using an indentation technique [7]. A total of 13 advanced ceramics and glass ceramic were used: 7 silicon nitrides (NC132, AS800 ('94), SN282, N3208 [4], AS800 ('99), NCX34, and NKK), one silicon carbide (α-SiC; Hexoloy), four aluminas (AD998 (Coors), AD998 [4], AD999 [4], and 96% alumina), and one glass ceramic (Pyroceram). Many properties of these materials have been evaluated previously by the authors [8].

3. RESULTS AND DISCUSSION

A summary of fracture toughness of a total of 13 advanced ceramics used in this work by both SEVNB and SEPB methods is presented in Figure 2. The figure compares the values of fracture toughness for each test material. As can be seen, agreement in fracture toughness between the two methods was good at lower fracture toughness, but poor at higher fracture toughness with a deviation becoming significant. This deviation can be seen more readily in terms of microstructural features -grain size and its shape- of test materials. Figure 3 depicts K_{Ic} ratio [=(K_{Ic} by SEVNB)/(K_{Ic} by SEPB)] with respect to a range of grain size. Fine grained ceramics and glass ceramic, such as α-SiC, AD999 alumina, NC132 and N3208 silicon nitrides, and Pyroceram, exhibited little difference in fracture toughness with K_{Ic} ratio close to unity. By contrast, coarse grained and elongated grained ceramics exhibited consistently lower K_{Ic} ratio ranging from 0.6 to 0.9 with an average of about 0.8. This indicates clearly that the two methods are in good agreement for fine-grained materials but in poor agreement with coarse or elongated grained materials.

It has been known that increase in fracture toughness for coarse or elongated grain-structured ceramics was due to mechanisms such as grain pullouts, grain bridging, and/or crack deflection,

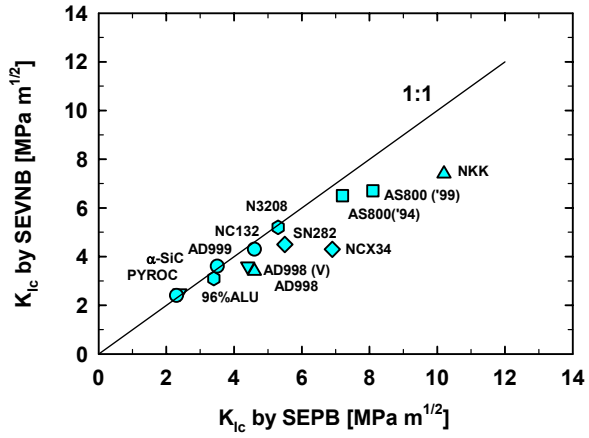

Figure 2. Fracture toughness determined by SEVNB and SEPB methods for various ceramics.

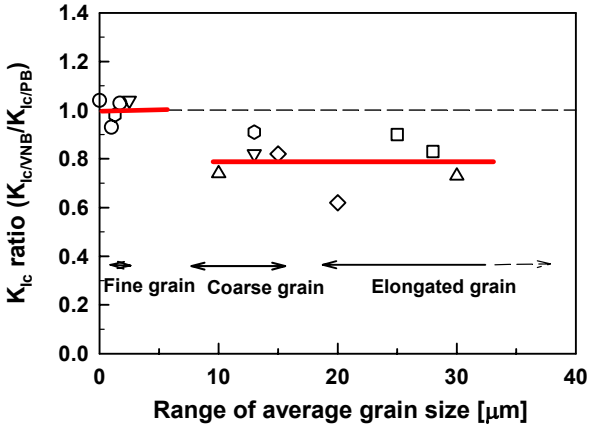

Figure 3. K_{Ic} ratio (=SEVNB/SEPB) with respect to range of grain sizes for various ceramics.

thereby generating crack closure stresses in the wake region of a crack. It has been also observed by the authors for a long time that these coarse and elongated grained ceramics reveled rising R-curve behavior with its degree depending on material [8]. Typical examples of R-curves of some materials evaluated by the indentation technique [7], are shown in Figure 4. Note that fine-grained NC132 silicon nitride (grain size <1 μm) and soda-lime glass showed flat R-curve behavior; whereas, coarse-grained AD998 alumina (grain size >10 μm) and elongated grained NKK and AS800 silicon nitrides (elongated grain size >20 μm) exhibited significant rising R-curves. The degree and size of grain elongation were greater in NKK than in AS800. The R-curve in Figure 4 was formulated using the following expression [7]

$$K_R = k[a]^m \tag{1}$$

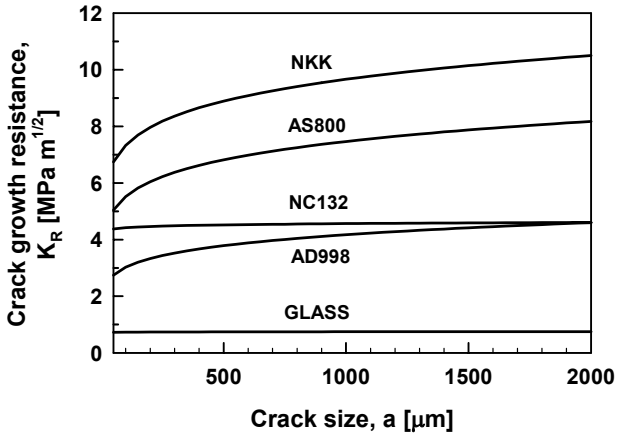

Figure 4. Crack growth resistance curves of some brittle materials, determined by indent techniques.

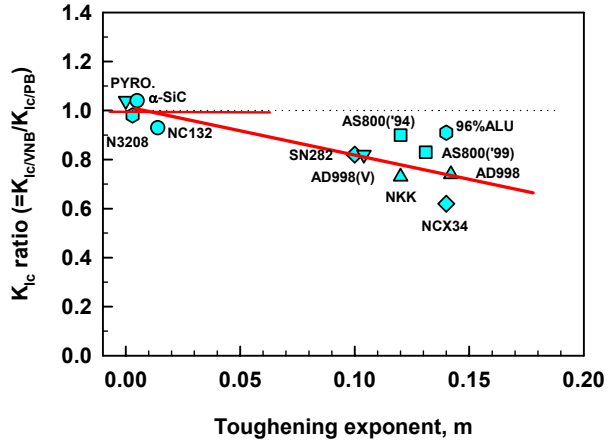

Figure 5. K_{Ic} ratio as a function of toughening exponent (*m*) for various ceramics.

where K_R is crack growth resistance, a is crack size (crack extension), k is a parameter, and m is toughening exponent. With the estimated m parameter for each test material, K_{Ic} ratio was plotted as a function of m, as shown in Figure 5. A clear trend can be seen from the figure such that K_{Ic} ratio remained close to unity for lower m values (m<0.02) but decreased appreciably (or dropped) at higher m values (m=0.1-0.14) with a following approximation

$$K_{Ic/SEVNB} / K_{Ic/SEPB} \approx -1.9\, m + 1 \qquad (2)$$

This, although not based on theoretical consideration, implies that the toughening exponent m could be an important parameter to quantify the degree of discrepancy in fracture toughness between the SEVNB and SEPB methods.

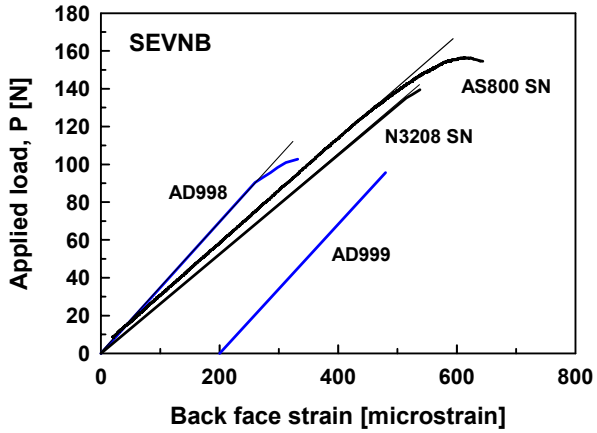

Figure 6. Typical examples of applied load-vs.-strain curves, determined by back-face strain gaging.

Typical load vs. back-face strain curves of SEVNB test specimens of several ceramics are shown in Figure 6 (Note that back-face strain was in compression but was expressed in tension for simplicity). Both fine-grained ceramics such as N3208 silicon nitride and AD998 alumina showed linearity in their load-vs.-strain curves. However, the coarse-grained AD998 alumina and the elongated-grained AS800 silicon nitride exhibited nonlinearity at the region close to the final fracture, resulting in stable crack growth prior to instability. This stable crack growth was determined analytically in conjunction with the results of Figure 6. The analytical solution was expressed as follows:

$$\alpha = 1 - [\frac{dP}{d\varepsilon}]^{1/2} f(L_o, L_i, b, W, E) \tag{3}$$

where α=a/W with a and W being crack size and specimen depth, P is applied load, ε is backside strain, and the function f depends on fixture spans, and width and elastic modulus of test specimen. The crack growth resistance K_R, corresponding to crack size (α) and applied load in the stable crack-growth region, was determined and the results are shown in Figure 7.

For N3208 and AD999 materials, no or negligible stable crack growth occurred. By contrast, AS800 material exhibited a significant stable crack growth, resulting in a considerable increase in fracture toughness by about 35 %. Also, AD998 material resulted in fracture-toughness increase by about 25 %. It is interesting to note from the figure that fracture toughness started from the values determined by the SEVNB method and ended up with the values determined by the SEPB method. This indicates that the discrepancy in fracture toughness between the two methods, particularly for coarse and elongated grained ceramics, is due to the fact that the calculation of K_{Ic} in SEVNB specimens did not consider the stable crack growth in their final crack sizes, which is not discernable from fracture surfaces in many cases. Although this extension by sable crack growth is small in the order of a few hundred micrometers and the measurement imposes a great difficulty, the effect on fracture toughness is still significant. Stable crack growth occurring via grain bridging and pullouts together with crack closure stresses acting in the wake region is the most plausible, physical explanation for the discrepancy in fracture toughness between the two methods.

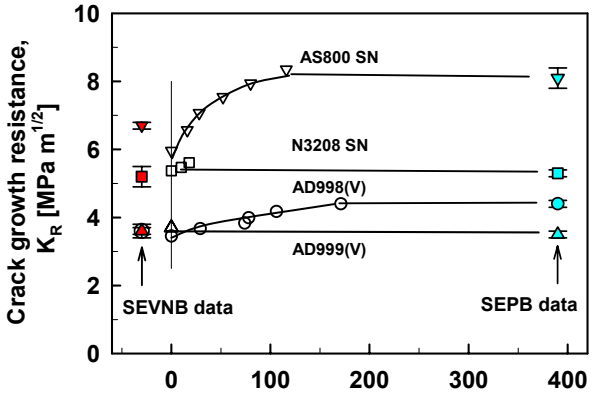

Figure 7. Crack growth resistance curves determined by back-face strain gaging based on the results in Figure 6.

4. CONCLUSIONS

The discrepancy in fracture toughness between the SEVNB and SEPB methods was due to R-curve behavior of ceramic materials, in which stable crack growth through grain pullouts and bridging takes place, imposing crack closure forces in the wake region of a propagating crack even for SEVNB specimens. The discrepancy increased for materials with stronger rising R-curve and was quantified with a toughening exponent (*m*). It is recommended that both SEVNB and SEPB methods be used together to evaluate fracture toughness of any materials unknown in microstructural features and R-curve behavior. Any elaborate method to monitor stable crack growth in SEVNB specimens is also recommended.

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