Micro-toughness of ZrB₂-based ceramics evaluated by ultra-shallow V-notch

Xinyuan Zhao, Anze Wang, Dazhao Liu, Huimin Yin, Zhiwei Xia, Changhao Xu and Peng Zhou

*School of Materials Science and Engineering, Nanjing Institute of Technology, Nanjing, P. R. China; †Institute of Intelligent Manufacturing Technology, Shenzhen Polytechnic, Shenzhen, P. R. China; ‡Institute of Metal Research, Chinese Academy of Sciences, Shenyang, P. R. China; §Jiangsu Key Laboratory of Advanced Structural Materials and Application Technology, Nanjing, P. R. China; ‡Anhui Key Laboratory of High-Performance Non-ferrous Metal Materials, Anhui Polytechnic University, Wuhu, P. R. China

ABSTRACT
Because of the essential difference between micro-toughness and macro-toughness, the indiscriminate application of deep notch tests may result in serious errors of micro-toughness associated with shallow cracks or flaws in ceramics. Here, the micro-toughness values of ZrB₂, ZrB₂-SiC, and ZrB₂-SiC-Grapite ceramics were successfully measured through ultra-shallow sharp V-notches (depth less than 40 μm, the ratio of notch depth to sample height less than 0.01) produced by nanosecond laser on the surface of bending bars for the first time. Results indicated that the micro-toughness values obtained from these three typical ceramics were significantly lower than the macro-toughness values measured from deep notches. Furthermore, conflicts may arise when comparing the micro- with macro-toughness between different materials, such as the macro-toughness of ZrB₂-SiC-G was larger than that of ZrB₂-SiC, while a contrary result was obtained for micro-toughness. Compared with the indentation method, improved accuracy of micro-toughness measurement could be obtained by this method.

1. Introduction
Fracture mechanics attempts to ensure the structural integrity of engineering materials which may contain cracks or flaws [1,2]. The usual approach is based on the measurement of a single parameter which is expected to quantify a lower bound of material toughness [3]. Fracture toughness is typically obtained from deeply notched bend specimens, which gives a macro-toughness value of fracture [4]. However, brittle fractures in structural ceramics are often associated with surface cracks or flaws of only several dozens of microns or so in depth [5], and the cracks would either be detected when shallow, or that flaws would not grow deep in a stable manner due to the nature of the service of the structure [6]. In this situation, the macro-toughness of the material is not directly pertinent to the fracture problem [7]. To make safety and realistic assessments of specific shallow cracks or flaws in structures, it is indispensable to carry out micro-toughness tests on specimens.

In order to measure the micro-toughness value, it is vitally important to ensure that the notch or the introduced flaw in the test specimen have the same orientation and microstructure with the crack or flaw in the material concerned. Surface crack in flexure (SCF) [8], indentation fracture (IF) [9], and shallow notch single edge V-notch beam SEVNB [10] methods are then of great advantage to the determination of micro-toughness value of ceramics. Carlton et al. [11] compared the fracture toughness values of three different purity of aluminum oxide (96%, 97.5%, and 99.5%) using SCF and deep notch SEVNB methods. Results indicated that the micro-toughness measured by SCF method was generally lower than that of SEVNB method (macro-toughness). The V-notch fabricated by picosecond laser in this study was very sharp (ρ < 10 μm, which was sharp enough for the accurate measurement of fracture toughness in aluminum oxide), and thus, it was reasonable to believe that the higher value in the SEVNB test was not caused by the blunt notch behavior, but reflected the essential difference between micro-toughness and macro-toughness.

Ponton and Rawlings [12] have also emphasized that the measured toughness (Kic) values of typical brittle materials (such as glass ceramics, aluminas, zirconias, etc.) are ranked as follows: SEVNB macro-toughness > IF micro-toughness, where SEVNB Kic was measured by deep notch. However, uncertainties of the test results of SCF and IF methods always arose due to the complex residual stress field produced by the indentation, and the results might be sample and purity dependent in SCF tests, also indentation force, accuracy of measured crack lengths, and calculation formula dependent in IF tests, eventually leading to a high statistical variation for micro-toughness. This discrepancy in micro-toughness results is unacceptable when performing any study where accuracy is important.
In this case, developing the shallow notch SEVNB method to test micro-toughness seems more reliable and promising. However, traditional notching methods (razor blade [13–15] and pop-in [16,17]) were difficult to control shallow but sharp notches in ceramics during preparation. Recently, laser micromachining technique has attracted great interest due to its superiority in efficient and high-precision processing. By using femtosecond laser, Zhao et al. [18] successfully introduced shallow notches with the depth of ~50 μm in Y-TZP ceramic and studied the effect of notch depth on the \( K_c \) value. Results clearly showed that the \( K_c \) (3.61 ± 0.21 MPa·m\(^{1/2}\)) measured by shallow notches was significantly lower than the deep notches results (4.42 ± 0.12 MPa·m\(^{1/2}\)). A similar trend was also found in ZrB\(_2\)-based ceramics and a prediction model of fracture toughness was established [19]. In contrast, in the reports of Turon-Vinas et al. [20,21], fracture toughness of 3Y-TZP and Si\(_3\)N\(_4\) measured by shallow and deep V-notches were almost the same. Note that the large amount of micro-cracks at the tip of notch generated during ultra-short pulsed laser ablation, these microcracks might induce the microcracking toughening mechanism [22,23], resulting in the overestimation of micro-toughness.

However, to our knowledge, studies in the area of micro-toughness measurement are still very limited at present. Some important problems remain unclear in micro-toughness measurement, such as notch depth effect and the relationship with macro-toughness. To help answer these important questions, the present paper compared the fracture toughness of three typical ceramics (ZrB\(_2\), ZrB\(_2\)-SiC and ZrB\(_2\)-SiC-G) using SEVNB samples with various notch depths fabricated by nanosecond laser micromachining technique. Furthermore, micro-toughness values acquired from the ultra-shallow V-notch tests were compared with the IF test results calculated by three classic models which were commonly used in ceramic studies.

2. Experimental procedures

2.1. Material and test samples

Commercial ZrB\(_2\) (mean particle size of 2 μm, purity > 99.5%, Northwest Institute for Non-ferrous Metal Research, China), SiC (mean particle size of 0.5 μm, purity > 99%, H.C. Starck, Germany) and graphite flake (mean diameter and thickness of 15 μm and 2 μm, respectively, purity > 99.0%, Qingdao Tiansheng Graphite Co., Ltd., Qingdao, China) were used to fabricate ZrB\(_2\), ZrB\(_2\)-20 vol.% SiC (ZrB\(_2\)-SiC) and ZrB\(_2\)-20 vol.% SiC-15 vol.% Graphite (ZrB\(_2\)-SiC-G) ceramics. Powders were ball-milled for 10 h at a speed of 200 rpm, and the uniformly mixed powder mixtures were hot pressed at 1950°C for 1.5 h under a uniaxial load of 30 MPa in Ar atmosphere. Detailed preparation procedures were described elsewhere [24]. The billets of ZrB\(_2\), ZrB\(_2\)-SiC and ZrB\(_2\)-SiC-G ceramics were then machined into 2 mm × 4 mm × 22 mm bars and the surfaces of these bars were polished to 0.5 μm finish with diamond slurries. All samples were carefully selected and the denser ones (relative densities between 90% and 95% for ZrB\(_2\), and larger than 99% for ZrB\(_2\)-SiC and ZrB\(_2\)-SiC-G, respectively) were picked out for micro- and macro-toughness tests. Grain sizes were estimated by the scanning electron microscopy (SEM, FEI Helios 600i, USA) micrographs of fracture surfaces of specimens.

2.2. Notch method

Nanosecond laser (Proton Laser Applications, Spain) with the wavelength of 1064 nm, pulse width of 10 ns, power of 1–10 W and scan speed of 1–50 mm/s was used to fabricate ultra-sharp V-notches on the center of the surfaces in test bars. By adjusting the laser notching parameters, that is, power, scan speed and notching times, V-notches with different depths (ranging from ~10 to ~423 μm) could be fabricated. It should be note that the notch depth had a certain maximum limit, owing to the fact that effective laser energy at the notch tip decreased with notch depth when laser processing position (away from the focal plane) moved down. Thus, in order to obtain deeper notches (> 400 μm), nanosecond laser method was recommended to be used in conjunction with the wire cutting method. All samples were annealed in vacuum at 1500°C for 30 min to eliminate the effect of the heat-affected zone around the notch tips. Morphologies of the V-notches were observed by the SEM.

2.3. SEVNB method

Micro- and macro-toughness were determined by the three-point SEVNB method (SFL-50KNAG, SHIMADZU, China) with a span of 16 mm and a cross head speed of 0.05 mm min\(^{-1}\). The toughness (\( K_c \)) value was obtained by using the following expression [25]:

\[
K_c = \frac{3P_{\text{max}}S_0}{2BW^2} \sqrt{\alpha}
\]  

(1)

\[
Y = \frac{1.1215\sqrt{\alpha}}{\beta^{3/2}} \left[ \frac{5}{8} \cdot \frac{5}{12} \cdot a^2 + \frac{1}{8} \cdot a^2 + 5a^2\beta^2 + \frac{3}{8} \exp\left(-\frac{6.1342a}{\beta} \right) \right]
\]

(2)

where \( P_{\text{max}} \) is the fracture load; \( S_0 \) is the span length (16 mm); \( B \) is the sample width (2 mm); \( W \) is the sample height (4 mm); \( a \) is the notch depth; \( \alpha \) is the relative notch depth defined by \( a/W \) and \( \beta = 1-\alpha \). The above equations are valid for any \( \alpha \) in the range of 0 < \( \alpha < 1 \).
In order to improve the accuracy of the $K_{IC}$ value, it should be noted that the notch depth was measured from the fracture surface instead of the side surface.

2.4. IF method

Hardness measurement was conducted using a Time Instrument HV-1000 Vickers Micro-hardness tester. Elastic modulus was measured in flexure using a three-point bending unit [26]. Indentations were conducted at the peak contact load of 30 kg for 30s on the polished faces of the specimens using a Vickers diamond pyramid. Obvious Radial-Median type crack system was observed in Figure 1b. Thus, the indentation micro-toughness of the material was evaluated by selecting three classic models (as presented in Table 1) commonly used in ceramic studies, where $P$ corresponds to the applied load of indentation, $c$ is the crack length, $H_v$ is the Vickers hardness and $E$ is the Young’s modulus.

The hardness and fracture toughness were averaged for at least five points.

Table 1. Selected equations for $K_{IC}$ measurements with Vickers indentations.

| No | Ref | Radial-Median type crack system |
|----|-----|---------------------------------|
| 1  | [27] | $K_{IC} = 0.0154 \cdot \left( \frac{P}{H_v c}\right)^{1/2} \cdot \frac{E}{\pi}$ |
| 2  | [28] | $K_{IC} = 0.0095 \cdot \left( \frac{P}{H_v c}\right)^{1/3} \cdot \frac{E}{\pi}$ |
| 3  | [29] | $K_{IC} = 0.022 \cdot \left( \frac{P}{H_v c}\right)^{1/3} \cdot \frac{E}{\pi}$ |

3. Results and discussion

The average grain sizes ($d$) of ZrB$_2$, ZrB$_2$-SiC and ZrB$_2$-SiC-G ceramics taken from the geometric analysis of the SEM image (Figure 2) are about 7.2, 2.6, and 2.7 μm, respectively. In order to obtain the actual fracture toughness ($K_{IC}$) value, the V-notch tip radius ($\rho$) may comply with the condition $3d \gg \rho$ [29,30]. Therefore, the V-notch tip radius must be at least smaller than 21.6, 7.8, and 8.1 μm, respectively, for ZrB$_2$, ZrB$_2$-SiC, and ZrB$_2$-SiC-G ceramics. Figure 3 shows the microscopic morphologies of the ultra-sharp V-notches with various depths. By using the nanosecond laser with the power of 5 W and the scan speed of 50 mm/s, an ultra-shallow V-notch with the depth of 51 μm can be fabricated. Partial magnification of Figure 3a, c indicates that the tip radius of the V-notch can be less than 2 μm, which is sharp enough to obtain the actual $K_{IC}$ value for these three ceramics. Furthermore, no microcracks are detected in the front of the notch tip.

Figures 4, 5 show the fracture surfaces of test samples after fracture toughness testing by SEVNB method. In Figure 4, the V-notches are only introduced by nanosecond laser notching method and the fracture surface can be divided into two regions: laser notching region and brittle fracture surface. The boundaries (highlighted with yellow dotted lines) between the two regions (A and B) are very clear and almost uniform from one side to the other side.
the depth of about 16 and 130 μm, respectively. Only a small amount of melting and recrystallizing layer, which has been proved to have no effect on the fracture toughness value [31,32], is observed without microcracks or other damages near the boundaries. Fracture surface of the V-notch fabricated by wire cutting combined with laser notching method, see Figure 5, can be divided into three regions: U-groove region, laser notching region and brittle fracture surface. The boundary between each region is also very clear and uniform.

Figure 6 shows the $K_c$ values of these three ceramics measured by different $a/W$. It is obvious that the measured values of $K_c$ by SEVNB method are closely correlated with the $a/W$. Note that the $K_c$ values increase rapidly with the $a/W$ values when $a/W$ is below a critical value, then remain constant at 2.89 ± 0.08, 3.34 ± 0.06 and 3.88 ± 0.10 MPa·m$^{1/2}$, which are the macro-toughness for ZrB$_2$, ZrB$_2$-SiC and ZrB$_2$-SiC-G ceramics, respectively. When $a/W$ reaches 0.6, the steep increase in $Y(a/W)$ leads to appreciable errors in calculating its value from the observed notch depth, resulting in an increase in $K_c$ [33]. Partial magnification of Figure 6 reveals that the toughness value almost remains unchanged when $a/W$ is less than 0.01, which is much lower than the definition of shallow notch toughness with $a/W$ values $< 0.45$ [10]. The $a/W$ value obtained here ($< 0.01$) is similar as that reported by Zhao [18] of 0.0125 for Y-TZP ceramic. In view of this situation, $a/W < 0.01$ is defined as ultra-shallow notch and the $K_c$ value obtained in this case is considered to be the micro-toughness.

Results indicate that the micro-toughness measured by ultra-shallow notches are associated with markedly lower values (2.12 ± 0.11, 2.75 ± 0.08, and 2.47 ± 0.09 MPa·m$^{1/2}$, respectively, for ZrB$_2$, ZrB$_2$-SiC, and ZrB$_2$-SiC-G ceramics) than the macro-toughness from deeply notched bend specimens. It can also be found that the
Figure 5. Fracture surface of ZrB$_2$-SiC-G test specimen notched by wire cutting combined with laser notching method, where three regions are shown: A (U-groove), B (V-notch by nanosecond laser) and C (brittle fracture surface).

Figure 6. The fracture toughness of ZrB$_2$-based ceramics with different $a/W$ measured by SEVNB method.
The macro-toughness of ZrB$_2$-SiC-G (3.88 ± 0.10 MPa·m$^{1/2}$) is larger than that of ZrB$_2$-SiC (3.34 ± 0.06 MPa·m$^{1/2}$), while a contrary result is obtained for micro-toughness. The main reason is that the micro-toughness tested by an ultra-shallow notch is determined by single-crystal cleavage energies (transgranular fracture) or grain-boundary energies (intergranular fracture), while the fracture toughness tested by a deep notch strongly depends on the microstructural influence and becomes representative of the polycrystalline aggregate. The prevailing interpretation of this tendency has been illustrated in Ref [34], which revealed that the fracture toughness increases in some systematic manner with crack or notch size. For ZrB$_2$-SiC-G ceramic, the multiple toughening mechanisms (such as crack deflection and branching, crack bridging [35] as well as pull-out of the graphite [36]) enhanced the fracture toughness at the macro-scale. However, at the micro-scale, the addition of graphite flakes is equivalent to the introduction of flaws, leading to the weak interfacial bonding and the reduction of fracture energy [36].

To our knowledge, grain-boundary sliding is also one of the potential reasons for the decrease of ceramic toughness. As reported in Ref [37], grain-boundary sliding of ZrB$_2$-SiC usually occurs at ultra-high temperature, so the effect of it on the toughness tests at room temperature could be negligible. The addition of graphite can promote the sliding of grain-boundaries, but the premise is that the grain size of the ceramic matrix is smaller than several micrometers, and the graphite platelet length is smaller than the grain-boundary length [38]. Given that the diameter of the graphite flakes (15 μm) in the ZrB$_2$-SiC-G ceramic is much larger than the average grain size (2.7 μm), grain-boundary sliding does not theoretically occur.

Figure 7. Comparison of $K_{IC}$ values among test methods for ZrB$_2$, ZrB$_2$-SiC, and ZrB$_2$-SiC-G ceramics.
Micro-toughness obtained by ultra-shallow V-notches is then compared with the indentation method. The measured hardness ($H_v$) of 10.1 ± 0.9, 18.5 ± 0.2, and 12.1 ± 0.6 GPa and the elastic modulus ($E$) of 375 ± 39, 443 ± 42, and 309 ± 21 GPa, respectively, for ZrB$_2$, ZrB$_2$-SiC, and ZrB$_2$-SiC-G ceramics, were used for the calculation of indentation micro-toughness. Figure 7 presents the fracture toughness measured by ultra-shallow notch, deep notch and indentations from different equations (No. 1 in Ref [27], and No. 2 and No. 3 in Ref [28], as listed in Table 1). It can be seen that all of the micro-toughness measured by the indentation method are lower than that of the deep notch SEVNB method, but are a little higher than that of the ultra-shallow V-notch method, accompanied by a larger dispersion (~10%). Note that the Vickers indentation test gives the fracture micro-toughness value in the sense that crack growth occurs under a constant load applied for a given time which induces an irreversible residual stress that operates both during loading and unloading [4]. The existence of residual stress could lead to the underestimation of the crack length, eventually resulting in the overestimation of micro-toughness. In the report of Nose [39], the $K_I$ values of Al$_2$O$_3$ ceramic evaluated by IF method are even much higher than that of single-edge-fatigue-cracked beam (SECB) method, in which the crack is deep and can be considered as an ideal sharp notch. In addition, indentation load, loading time, formula used and other factors could also lead to the different micro-toughness results and high deviations (sometimes even up to 48% [40]). In contrast, the ultra-shallow notch has a less deviation of micro-toughness measurement (~5%) compared with the indentation method.

4. Conclusions

Micro-toughness is one of the most important parameters in evaluating ceramic materials and material selection for specific engineering uses, especially in making safety and realistic assessments of specific shallow cracks or flaws in structures. In order to avoid the overestimation of micro-toughness, notches in the test samples must be shallow enough. In this study, nanosecond laser was successfully used to fabricate sharp V-notch with ultra-shallow depth (< 20 μm) in ZrB$_2$, ZrB$_2$-SiC and ZrB$_2$-SiC-G ceramics. Results indicate that the fracture toughness values of these ceramics tested by shallow notches show a strong dependence to the notch depth. To obtain the actual micro-toughness value for ZrB$_2$-based ceramics, we recommend that the ratio of notch depth to sample height ($a/W$) should not exceed 0.01. It is worth noting that the micro-toughness is generally lower than the macro-toughness, however, there is no necessary connection between them. Furthermore, conflicts may arise when comparing the micro- and macro-toughness values between different materials, for example the macro-toughness of ZrB$_2$-SiC-G is larger than that of ZrB$_2$-SiC, while a contrary result is obtained when it comes to micro-toughness. Overall, the present study on ultra-shallow SEVNB method can provide a lower bound estimate of toughness in ZrB$_2$-based ceramics, and improved accuracy can be obtained by this method compared with indentation method.

Acknowledgments

Financial support was provided by the Natural Science Foundation of Jiangsu Province (No. BK20201040), the China Postdoctoral Science Foundation (No. 2022M713216), the Opening Project of Jiangsu Key Laboratory of Advanced Structural Materials and Application Technology (No. AM2002108), Post-doctoral fund of Shenzhen Polytechnic (No. 4103-6021330011K0) and the Open Research Fund of Anhui Key Laboratory of High-Performance Non-ferrous Metal Materials (No. KP10000005).

Disclosure statement

No potential conflict of interest was reported by the author(s).

Funding

This work was supported by the Natural Science Foundation of Jiangsu Province [BK20201040].

References

[1] Hancock JW. A two parameter approach to the fracture behaviour of short cracks. Shallow Crack Fracture Mechanics, Toughness Tests and Applications; 1992.
[2] Lawn B. Fracture of brittle solids. Maryland: Cambridge university press; 1993.
[3] Launey ME, Ritchie RO. On the fracture toughness of advanced materials. Adv Mater. 2009;21:2103–2110.
[4] Ponton CB, Rawlings RD. Vickers indentation fracture toughness test Part 2 application and critical evaluation of standardised indentation toughness equations. Mater Sci Tech. 1989;5:961–976.
[5] Dawes MG, Gordon JR. The requirement for shallow crack fracture toughness tests. UK: Cambridge; 1992.
[6] Mathews JR, Hyatt CV, KarisAllen KJ. A general discussion of short crack fracture. Shallow Crack Fracture Mechanics, Toughness Tests and Applications. 1992;16.
[7] Evans AG. Structural and microstructural design in brittle materials. Berkeley: Springer; 1980.
[8] Quinn GD, Swab JJ. Fracture toughness of glasses as measured by the SCF and SEPB methods. J Eur Ceram Soc. 2017;37:4243–4257.
[9] Quinn GD, RC B. On the Vickers indentation fracture toughness test. J Am Ceram Soc. 2007;90:673–680.
[10] Dawes MG. Shallow crack fracture mechanics toughness tests and applications: first international conference. Sawston, Cambridge: Woodhead Publishing; 1993.

[11] Carlton HD, Elmer JW, Freeman DC, et al. Laser notching for reliable fracture toughness testing. J Eur Ceram Soc. 2016;36:227–234.

[12] Ponton CB, Rawlings RD. Vickers indentation fracture toughness test Part 1 review of literature and formulation of standardised indentation toughness equations. Mater Sci Tech. 1989;5:865–872.

[13] Damani R, Gstrein R, Danzer R. Critical notch-root radius effect in SENB-S fracture toughness testing. J Eur Ceram Soc. 1996;16:695–702.

[14] Fett T. Influence of a finite notch root radius on fracture toughness. J Eur Ceram Soc. 2005;25:543–547.

[15] Kübler J. Fracture toughness of ceramics using the SEVNB method: preliminary results. Ceram Eng Sci Proc. 1997;18:155–162.

[16] Miyazaki H, Hyuga H, Hirao K, et al. Comparison of fracture resistance as measured by the indentation fracture method and fracture toughness determined by the single-edge-precracked beam technique using silicon nitrides with different microstructures. J Eur Ceram Soc. 2007;27:2347–2354.

[17] Miyazaki H, Yoshizawa Y, Hirao K, et al. Round-robin test on the fracture toughness of ceramic thin plates through modified single edge-precracked plate method. J Eur Ceram Soc. 2016;36:3245–3248.

[18] Zhao W, Peng C, Lv M, et al. Effect of notch depth on fracture toughness of Y-TZP and determination of its actual value. Ceram Int. 2015;41:869–872.

[19] Wang AZ, Wang YZ, Zhang X, et al. On the estimation and modeling of fracture toughness in structural ceramics in a simple way. Theor Appl Fract Mech. 2019;103:102273.

[20] Turon-Vinas M, Anglada M. Fracture toughness of zirconia from a shallow notch produced by ultra-short pulsed laser ablation. J Eur Ceram Soc. 2014;34:3865–3870.

[21] Turon-Vinas M, Anglada M. Assessment in Si3N4 of a new method for determining the fracture toughness from a surface notch micro-machined by ultra-short pulsed laser ablation. J Eur Ceram Soc. 2015;35:1737–1741.

[22] Xia K, Langdon TG. The toughening and strengthening of ceramic materials through discontinuous reinforcement. J Mater Sci. 1994;29:5219–5231.

[23] Hu XZ, Lutz EH, Swain MV. Crack-tip-bridging stresses in ceramic materials. J Am Ceram Soc. 1991;74:1828–1832.

[24] Wang AZ, Hu P, Zhang XH, et al. Accurate measurement of fracture toughness in structural ceramics. J Eur Ceram Soc. 2017;37:4207–4212.

[25] Munz D, Fett T. Ceramics: mechanical properties, failure behaviour, materials selection. 1st ed. Berlin, Heidelberg: Springer-Verlag; 1999.

[26] Scherrer SS, De Rijk WG. The fracture resistance of all-ceramic crowns on supporting structures with different elastic moduli. Int J Prosthodont. 1993;6:462–467.

[27] Anstis GR, Chantikul P, Lawn BR, et al. A critical evaluation of indentation techniques for measuring fracture toughness: I. direct crack measurements. J Am Ceram Soc. 1981;64:533–538.

[28] Laugier MT. The elastic/plastic indentation of ceramics. J Mater Sci Lett. 1985;4:1539–1541.

[29] Fett T. Estimated stress intensity factors for semi-elliptical cracks in front of narrow circular notches. Eng Fract Mech. 1999;64:357–362.

[30] Fischer H, Waindich A, Telle R. Influence of preparation of ceramic SEVN8 specimens on fracture toughness testing results. Dent Mater. 2008;24:618–622.

[31] Zhao W, Cui JP, Rao PG. Effect of molten zone ablated by femtosecond lasers on fracture toughness of Si3N4 measured by SEVN8 method. J Eur Ceram Soc. 2018;38:2243–2246.

[32] Zhao W, Cui JP, Rao PG. Effect of thermal stress induced by femtosecond laser on fracture toughness of fine-grained alumina. J Aust Ceram Soc. 2019;55:575–578.

[33] Sakai M, Bradt KC. Fracture toughness testing of brittle materials. Int Mater Rev. 1993;38:53–78.

[34] Cook RF, Lawn BR, Fairbanks CJ. Microstructure-strength properties in ceramics: I. effect of crack size on toughness. J Am Ceram Soc. 1985;68:604–615.

[35] Zhou S, Wang Z, Zhang W. Effect of graphite flake orientation on microstructure and mechanical properties of ZrB2-SiC-graphite composite. J Alloys Compd. 2009;485:181–185.

[36] Zhang Z, Wang Z, Sun X, et al. Effect of graphite flake on the mechanical properties of hot pressed ZrB2-SiC ceramics. Mater Lett. 2008;62:4360–4362.

[37] Talmy IG, Zaykoski JA, Martin CA. Flexural creep deformation of ZrB2/SiC ceramics in oxidizing atmosphere. J Am Ceram Soc. 2008;91:1441–1447.

[38] Sheinerman AG, Morozov NF, Gutkin MY. Effect of grain boundary sliding on fracture toughness of ceramic/graphene composites. Mech Mater. 2019;137:103126.

[39] Nose T, Fujii T. Evaluation of fracture toughness for ceramic materials by a single-edge-precracked-beam method. J Am Ceram Soc. 1988;71:328–333.

[40] Fischer H, Marx R. Fracture toughness of dental ceramics: comparison of bending and indentation method. Dent Mater. 2002;18:12–19.