Standardization of the Laser Notching Method for Measuring Fracture Toughness in Structural Ceramics

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Research Article

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Posted Date: January 25th, 2021

DOI: https://doi.org/10.21203/rs.3.rs-152425/v1

Standardization of the laser notching method for measuring fracture toughness in structural ceramics

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Abstract

The single-edge V-notched beam (SEVNB) method based on the laser notching approach can effectively overcome the shortcoming of time-consuming and avoid large errors in traditional fracture toughness measurement ways, nevertheless the laser notching method has not yet been standardized. Taking oxide (ZrO_2 and Al_2O_3), carbide (SiC), nitride (Si_3N_4) and boride (ZrB_2-based) ceramics as the research objects, this paper systematically discussed the effects of notch tip sharpness, notch depth and equivalent notch angle on the measured value of fracture toughness, thereby clearly defined the range of these parameters that required for measuring the fracture toughness accurately. Furthermore, in order to give full play to the advantages of the laser notching method, the feasibility of sample miniaturization was also discussed. This study could provide important data reference and theoretical basis for the standardization of laser method in the near future.

Keywords: Fracture toughness; Structural ceramics; Notch tip radius; Notch depth; Equivalent notch angle

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1. Introduction

Fracture toughness is one of the key and intrinsic material properties that is known as the critical stress intensity factor (K_c) [1]. As ceramics mostly fail in mode I, which is the most common mode for laboratory testing, the K_c in mode I is referred to as K_{Ic}, which represents the fracture toughness corresponding to how much energy a material can absorb before catastrophic failure occurs under this mode. Accurate measurement of K_{Ic} is thus of great significance for the design, material selection and reliability evaluation of ceramic materials [2].

Under the unremitting efforts of scientists, numerous of standards for fracture toughness tests have been implemented worldwide, such as ASTM C1421-16 [3], ASTM E1820-13 [4], ASTM E1290-08 [5], ISO 15732-2003 [6], ISO 24370-2005 [7], ISO 23146-2012 [8], GB/T 23806-2009 [9] and DIN EN 14425-3 [10]. The recommended test methods in each standard are different, mainly including: single-edge V-notched beam (SEVNB) [11], single-edge precracked beam (SEPB) [12], chevron notch beam (CNB) [13], surface crack in flexure (SCF) [14], crack-tip opening displacement (CTOD) [15] methods, etc., as shown in Table 1.

Despite there is a wide range of methods to evaluate a ceramic's fracture toughness and the selection can be material and resource dependent, each one has obvious shortcoming. The SCF test needs fractographic expertise and careful grinding to remove residual stress, thus leading to a high randomness in its results [16]. The CNB method is generally preferred over the SCF method because it produces more consistent results if the notches are properly fabricated. However, the notch formation is difficult, which requires special machining fixture, and the stable crack growth in the notch plane is hard to detect [17]. The disadvantage of the CTOD specimen is that it is costly and difficult to machining owing to its relatively large and complex geometries [18]. SEPB method overcomes the drawback of single-edge notched beam (SENB) method, but the reliable introduction of a real crack into the ceramic sample is very difficult and the crack depth is uncontrollable [19,20].

By contrast, SEVNB, as an alternative to SEPB, is essentially a derivative of SENB method, replacing real crack with sharp V-shaped notches, which is an

convenient and frequently used method. Meanwhile, it has been recognized as having great advantages in specimen preparation, testing process, data accuracy, consistency, and reproducibility. In addition, SEVNB test procedure could also be easily modified toward the assessment of fracture toughness under shear loading via placing an eccentric notch on a testing specimen [17,21].

Table 1 Advantages and disadvantages of using fracture toughness methodologies in various testing standards

Testing standards	**Areas**	**Method**	**Advantages**	**Disadvantages**
ISO 23146-2012	International	SEVNB	• Relatively easy specimen fabrication • Low cost fixture • Does not require advanced fractography skills	• Difficult to achieve adequate V-notch fabrication
ASTM E1820-13	USA	SEVNB		
		CTOD	• A relatively long crack length is available and relatively stable crack extension occurs under displacement controlled loading using a high stiffness testing machine which is ideal for subcritical crack growth and R-curve measurements	• A relatively complex specimen geometry which makes machining difficult and costly • The delicate precracking procedure, where some type of crack arrestor attachment is required for most brittle ceramics
ASTM E1290-08	USA	CTOD		
DIN EN 14425-3	Germany	CNB	• Not necessary to measure a precrack	• Notch formation is difficult • Needs special machining fixtures • Requires stable crack growth in the notch and stable crack growth may be difficult to detect • Crack needs to follow notch plane
ISO 24370-2005	International	CNB		
ASTM C1421-16	USA	CNB		
		SCF	• Sharp and clinically-sized precracks • R-curve sensitive	• Needs fractographic expertise • Careful grinding to remove residual stress • Needs mirror polished surface
		SEPB	• Produces sharp precracks • R-curve sensitive	• Very difficult to produce real precrack (involves additional fixture and crack detection)
ISO 15732-2003	International	SEPB		
GB/T 23806-2009	China	SEPB		

It is noteworthy that preparing sharp enough V-notches on ceramic specimens is the prerequisite and also the difficulty for reliable fracture toughness assessment in SEVNB test. As we all know, apparent K_{Ic} values that are calculated from the maximum loads decrease with decreasing notch radius until some critical notch tip radius ρ_c, and the apparent K_{Ic} eventually become constant at the true value [18]. The ρ_c has been evident that is strongly dependent on the microstructure of the material.

However, there is no universal standard to determine the ρ_c value. Generally, the V-notch tip radius (ρ) should satisfy the condition that the ρ is less than three times the average grain size in order to obtain the actual fracture toughness value [22]. For example, ρ_c value of very fine-grained yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) ceramic with an average grain size of 0.5 μm appears to be about or even less than 1.5 μm, which in turn gives the theoretical upper limit of the sharp V-notch. Further clarification of the relationship between ρ_c and other microstructure characteristics of ceramics will help improve the measurement effectiveness of the SEVNB method.

In the last few decades, V-notch was often introduced by micro-milling [23] and/or razor blade sliding [22,24,25]. It is not only a time-consuming procedure, but also uncontrollable in keeping the same V-notch tip radius on both sides of the test specimens. More importantly, fabricating a sharp V-notch with the tip radius less than 5~10 μm still remains a difficult task. Even if a sharp V-notch is finally acquired, the 5 μm radius may still be insufficient for measuring the fracture toughness of fine ceramics, so that an overestimation of fracture toughness would come up due to the notch passivation effect [26].

Recently, laser micromachining techniques (including nanosecond laser [26], picosecond laser [27] and femtosecond laser [28-30]) have aroused scholars' great interests due to its high machining accuracy as well as efficiency, and have been successfully utilized to introduce sharp V-notches with tip radius less than 1 μm (0.5 μm [26,28] and 0.8 μm [23]) in various materials (e.g. ZrO_2, Si_3N_4, ZrB_2-SiC, etc.) so far. Although the laser method will inevitably lead to physical changes of the material (such as remelting and recrystallization), several previous studies have shown that the molten zone at the V-notch tip is generally less than 2 μm, which is ignorable during fracture toughness tests [31,32]. Furthermore, Zhao *et al.* [33] have revealed that the effect of thermal stresses induced by laser can also be ignored after comparing the fracture toughness value of the fine-grained alumina before and after annealing. Apart from these, the laser method has many other exciting advantages: (*i*) excellent efficiency (sharp V-notches are easy to prepare and only takes a few seconds); (*ii*) flat

bottom of the V-notch and good consistency of the notch depth on both sides of the sample and (*iii*) the plane of the final crack measured from the tip of the V-notch always parallel to both the test specimen dimensions width and thickness within 5°. Based on these significant advantages, many scholars have pointed out that the laser method is a very promising standardized method for measuring fracture toughness.

At present, there are still many challenges in the popularization and application of laser method, including: (*i*) laser notching is generally based on experience, while lack of available criteria to point out how sharp the V-notches should be for different ceramics with various compositions and microstructures; (*ii*) based on the current test standards, the notch depth should be in the order of millimeters, far beyond the laser notching depth (tens to hundreds of microns). Therefore, the laser method should be utilized in combination with the traditional methods, which leads to an equivalent notch angle in the compound notch (U-groove+V-notch) [11]. It is urgent to clarify the reasonable range of equivalent notch angle, that is, to define the relationship between the V-notch depth and the U-groove width; (*iii*) it is essential to fully understand the relationship between relative notch depth (notch depth/sample thickness) and the fracture toughness measurement values of different ceramics, so as to formulate the reasonable notch depth range; and (*iv*) the corresponding improvement of specimen size, especially the feasibility of small and thin specimens.

In response to the above problems, taking a variety of typical structural ceramics (including commercial 5Y-TZP, Si_3N_4, SiC and Al_2O_3 ceramics as well as self-prepared ZrB_2-based ceramics) as the study objects, the relationship between fracture toughness measurement values and notch tip radius, notch depth, equivalent notch angle as well as sample size were systematically studied.

2. Experimental procedures

2.1 Material and test samples

5 mol% yttria-stabilized tetragonal zirconia polycrystal (5Y-TZP), SiC and Al_2O_3 ceramics were provided by Zhuhai Jiawei Ceramic Technology CO., LTD. Si_3N_4 ceramics were purchased both from Zhuhai Jiawei Ceramic Technology CO., LTD. and Zhuhai Yuebojia New Material CO., LTD., and we name it Si_3N_4(J) and Si_3N_4(Y),

respectively. ZrB_2, ZrB_2-20 vol.% SiC (ZrB_2-SiC) and ZrB_2-20 vol.% SiC-15vol.% Graphite (ZrB_2-SiC-Graphite) ceramics were fabricated by hot-pressing method, and the detailed preparation procedures were described in our previous article [26]. Substrates were cut into the test pieces with the size of $2 \times 4 \times 22$ mm^3 and $3 \times 4 \times 40$ mm^3 in accordance with ASTM E1820-13 [4] and ISO 15732-2003/GB/T 23806 [6,9], respectively, to study the effects of notch tip radius, notch depth and equivalent notch angle on the measured fracture toughness value. Moreover, non-standard samples with the sizes of $2 \times 1 \times 16$ mm^3 and $2 \times 2 \times 16$ mm^3 were also prepared from the same bulk for the comparison of the sample size effect. The fracture surfaces of all specimens were checked by the scanning electron microscopy (SEM, Merlin Compact, Carl Zeiss AG), and the micrographs are shown in Fig. 1.

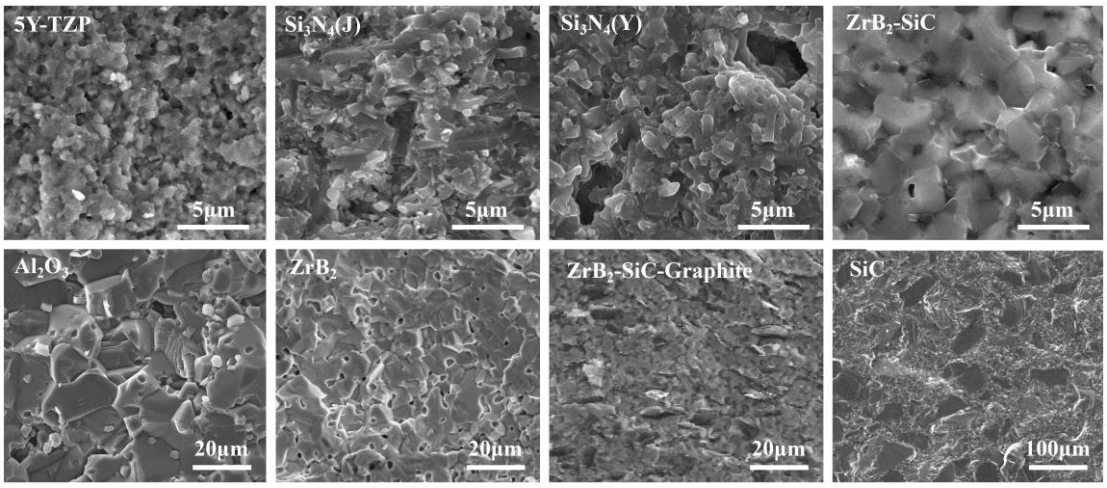

Fig. 1. SEM images of fracture surfaces of test samples.

2.2 Notching methods

U-grooves with different widths and depths on the $B \times S_0$ surfaces in non-conductive (5Y-TZP, Si_3N_4(J), Si_3N_4(Y), SiC and Al_2O_3) and conductive (ZrB_2, ZrB_2-SiC and ZrB_2-SiC-Graphite) ceramic samples were fabricated using diamond wheels and wire cutting machine, respectively. To get sharper notches with different tip radius, V-notches were polished along the prefabricated U-grooves using razor blades sprinkled with the 1 to 20 μm diamond pastes. However, the notch tip radius (ρ) prepared by the above-mentioned traditional methods was difficult to reach 10 μm.

In order to introduce sharper V-notches, here, nanosecond laser (DX-FM20, Nanjing Dingxin Electromechanical Equipment Co., Ltd., China) with the wavelength

of 1064 nm, pulse width of 10 ns, power of 1-20 W and scan speed of 10-50 mm·s^{-1} was used for multiple processing on the center of the surfaces in test bars. Typical sharp notch on ceramic sample is presented in Fig. 2. By adjusting the laser notching parameters, i.e., power, scan speed and notching times, sharp V-notches with different depths (ranging from 10 to 500 μm) could be achieved. For deeper notches, laser method could be utilized in conjunction with traditional notching method, see Fig. 3. In this case, it should be noted that the equivalent notch angle (θ) will change with the width of the U-groove and the depth of the V-notch.

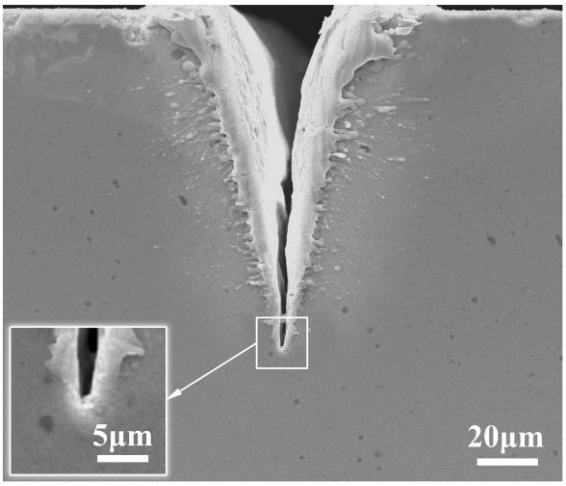

Fig. 2. SEM image of 5Y-TZP ceramic bar with an ultra-sharp V-notch. The enlarged view shows that the V-notch tip radius is ~0.7 μm and no microcrack is found near the V-notch root.

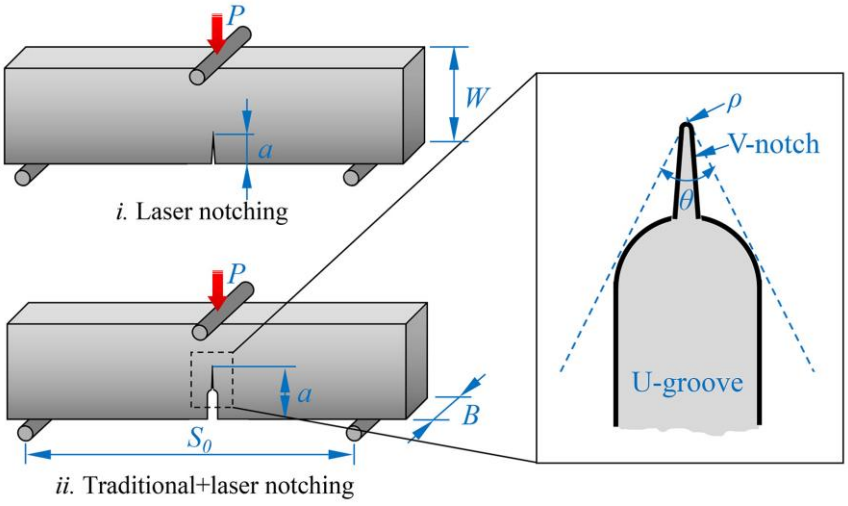

Fig. 3. Schematic of the SEVNB test specimens with sharp notches.

2.3 Fracture toughness tests

Fracture toughness was determined by the three-point SEVNB method (SFL-50KNAG, SHIMADZU, China) with a cross head speed of 0.05 mm·min^{-1}. The fracture toughness (K_{Ic}) of standard-sized specimens were calculated with reference to the corresponding standards [4,6], while the K_{Ic} value of non-standard samples were calculated by using the following expression [34]:

$$K_{Ic} = \frac{3P_{\max}S_0}{2BW^2} Y\sqrt{a} \tag{1}$$

$$Y = \frac{1.1215\sqrt{\pi}}{\beta^{3/2}}\left[\frac{5}{8} - \frac{5}{12}\alpha + \frac{1}{8}\alpha^2\beta^6 + \frac{3}{8}\exp(-\frac{6.1342\alpha}{\beta})\right] \tag{2}$$

where $P_{\max}$ is the fracture load; S_0 is the span width; B is the sample width; W is the sample height; a is the notch depth; α is the relative notch depth defined by a/W and $\beta = 1-\alpha$. All of the notch depths were carefully measured by SEM examinations of the fractured cross sections of the specimens.

3. Results and discussion

3.1 Effect of notch tip radius

According to the Griffith's theory, the critical stress intensity factor (K_c) remains unchanged, which can be regarded as the fracture toughness (K_{Ic}), when ρ is within the critical value (ρ_c), and is proportional to $\rho^{0.5}$ when ρ exceeds the threshold level, as shown by the gray dotted line in Fig. 4a. However, it exhibits different degrees of error on this criterion for different ceramics, which is reflected in the change of slope [26]. To thoroughly understand the relationship between K_c and ρ, the experimental data were analyzed in double logarithmic coordinates, and the data in the literature were redrawn at the same time for comparison, as depicted in Fig. 4b-f. The apparent K_{Ic} values of the fifteen kinds of ceramics increase linearly with the notch tip radius when $\rho > \rho_c$. Linear regression is used to obtain the best fit for the data and the slope is in the range of 0.1-0.5. This indicates that the fracture toughness (K_{Ic}) of structural ceramics is proportional to ρ^n, where n is normally less than 0.5 and strongly depends on the material itself. The greater the value of n, the more sensitive the material to the notch tip radius. Furthermore, a blunt notch may cause some errors in the comparison of K_{Ic} between different kinds of materials. For example, the K_{Ic} of Al_2O_3(T) is larger

than that of Al_2O_3(B) in the case of ρ < 200 μm, while a contrary result is obtained when ρ > 200 μm, as can be seen in Fig. 4f. Similar results can also be detected when comparing ZrB_2-SiC with ZrB_2-SiC-Graphite ceramics [26].

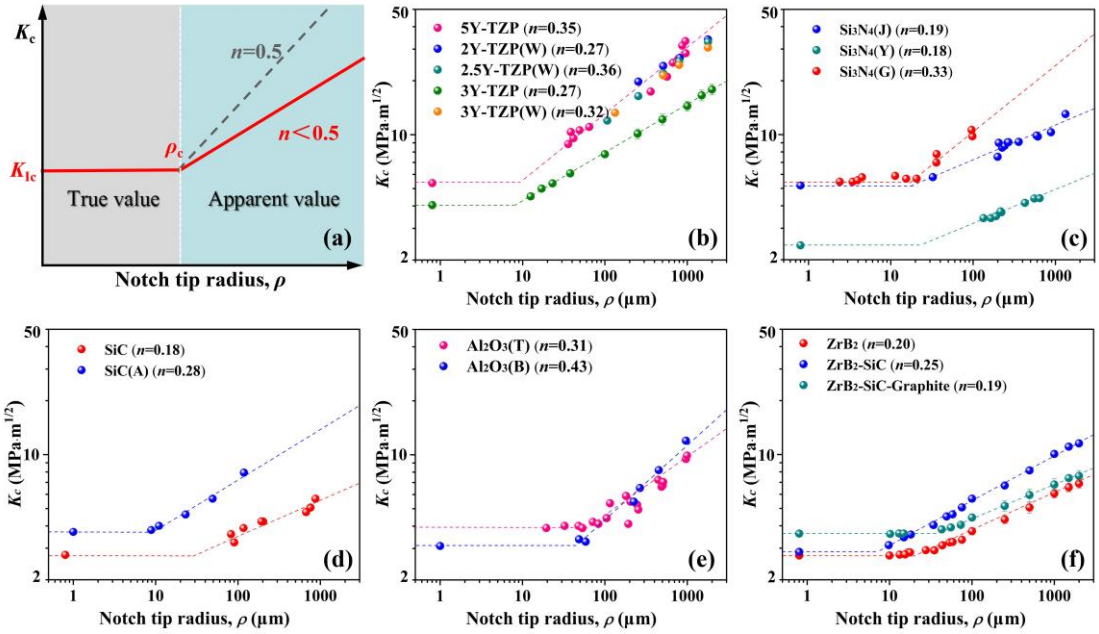

Fig. 4. Variation of the critical stress intensity factor (K_c) with notch tip radius (ρ). (a) Schematic diagram and test results of (b) yttria-stabilized zirconia, (c) silicon nitride, (d) silicon carbide, (e) alumina as well as (f) ZrB_2-based ceramics in double logarithmic coordinates.

In the measurement of fracture toughness via SEVNB method, the relationship between K_c, ρ_c and n value is very noticeable: (*i*) For the same type of material, the fracture toughness is not necessarily related to the slope or the critical notch tip radius. The slope of 0.28 in SiC(A) ceramic with high fracture toughness (3.70 MPa·$m^{1/2}$) is higher than that of SiC with relative low fracture toughness, but the ρ_c of SiC(A), less than 10 μm, is only ~1/3 of that of SiC, as illustrated in Fig. 4d and Table 2, which means that the V-notch required for the accurate measurement of fracture toughness in SiC(A) is sharper. On the contrary, alumina with high fracture toughness corresponds to a lower slope (n) and a higher critical notch tip radius (ρ_c), see Fig. 4e. (*ii*) For materials with similar ρ_c and n values, the difference in fracture toughness values may also be significant. For example, in Fig. 4c, even though the ρ_c and n of Si_3N_4(J) and Si_3N_4(Y) are almost the same as ~20 μm and ~0.18, respectively, the fracture

toughness values of the two are more than doubled (5.20 and 2.41 MPa·m$^{1/2}$, respectively). (*iii*) Even though the fracture toughness are similar, the material's sensitivity to the notch may vary greatly, which reflected in the notable difference of slope *n*, such as Si_3N_4(J) and Si_3N_4(G), the fracture toughness of the former are close to that of the latter (~5 MPa·m$^{1/2}$), but the notch sensitivity and the *n* value of Si_3N_4(J) decrease obviously which may owing to the whisker toughening by rod-like silicon nitride, as depicted in Fig. 2.

Although the relationship between the K_c, ρ_c and *n* value is very complicated, the most important concern is how sharp the V-notch tip radius (ρ) is enough to obtain the actual fracture toughness value. It is generally accepted that ρ_c seems to be strongly dependent on the microstructure of the material [18,35], and the previous theoretical calculation formulas can be expressed as:

$$\rho_c \approx \frac{4Y_0^2}{[\tanh^{-1} X]^2}\delta a \tag{3}$$

in Damani model [36] and

$$\rho_c = [\frac{x^* K_{Ic}^2}{8.8E\sigma_Y \varepsilon_f(R_\beta)}]^{1/2} \tag{4}$$

in Malkin model [37], respectively, where δa is the size of the flaw that caused the fracture initiation, Y_0 is 1.12 for a through-thickness edge crack, X is between 0.9 and 0.95, K_{Ic} is fracture toughness, σ_Y is yield strength in tension, E is elastic modulus, x^* is the size of the constant strain zone at the notch root, and $\varepsilon_f(R_\beta)$ is tensile fracture strain. In Eq. (3), the notch-tip small cracks are assumed to be related to some characteristic feature of the microstructure so δa is depended on the basis of microstructural studies. However, no obvious suitable microstructural feature is found at present. In Eq. (4), the accuracy of the ρ_c and K_{Ic} values are interdependent and the x^* value that describes the microscopic properties of the material is very difficult to obtain.

In view of these limitations, experimental estimation of the ρ_c in ceramic is more popular. Previous studies have pointed out that the ρ_c is positively correlated with the average grain size (d_{50}) and appears to be less than 10 μm for some fine grain size

ceramics [18], such as polycrystalline alumina (d_{50} is ~1 μm and ρ_c is ~9 μm) [38], zirconia (d_{50}＜1 μm and ρ_c＜10 μm) [36] and hot-pressed silicon nitride (d_{50}＜3 μm and ρ_c＜15 μm) [36]. Some scholars believe that the ρ should comply with the condition that the ρ should be less than three times the average grain size ($3d$) [22], however, there is a lack of experimental verification. Here, the critical notch tip radius (ρ_c) can be deduced according to the linear law in Fig. 4. It is found that $3d$ is often much lower than the ρ_c for fine-grained ceramics (e.g., ρ_c value of very fine-grained 5Y-TZP ceramic with an average grain size of 0.5 μm appears to be about or even less than 1.5 μm, much less than the critical value of 9.41 μm obtained by experiments, see Table 2), and if the excessively strict notch sharpness requirements of $3d$ are continued, it will be extremely difficult to introduce sharp enough notches into fine-grained ceramics, which are both hard and brittle, by traditional notching method.

As reported by Damani *et al*. [36], the magnitude of ρ_c is proportional to the size of the critical fracture-initiating flaw, corresponding to the the critical flaw size (a_c), which reflects the sensitivity of the material to stress concentration. Here, we compared the a_c and the ρ_c subsequently, wherein the a_c of these ceramics were calculated by the following formula [39]:

$$a_c \approx K_{Ic}^2/(\pi\sigma_0^{\ 2}) \tag{5}$$

where σ_0 is the original strength and the K_{Ic} value is obtained from sharp notched SEVNB method. As listed in Table 2 below, the a_c of most ceramics are very close to their ρ_c, while the a_c values of some ceramics with R-curve behaviour are even slightly lower than that of the ρ_c. For instance, ZrB_2-SiC-G ceramic, which exhibits significant R-curve behavior caused by the graphite toughening phase, as reported in Ref. [40], has a larger ρ_c of 36.17 μm than the a_c of 25.56 μm. The ρ_c of 45.43 μm for coarse-grained alumina, Al_2O_3(B), which could be accompanied by obvious crack propagation resistance [41], is also much greater than its a_c of 24.97 μm. The above results are consistent with the results of Fett [42], which indicate that the existence of R-curve behaviour imply higher value of the critical notch tip radius required in SEVNB specimens for a reliable assessment of the fracture toughness.

Therefore, regardless of whether the ceramic has R-curve characteristics, a_c can be regarded as a new criterion for making sharp notches, i.e., the accuracy of measured value of fracture toughness can be assured as long as the notch tip radius, ρ, is less than a_c. Although the calculation of a_c relies on the measurement of K_{Ic}, as can be seen in Eq. (5), the lower limit of the critical notch tip radius in ceramics, ρ_c, (corresponding to the a_c) can be obtained through extreme case assumptions. Generally, the fracture toughness value of ceramics is not less than 2 MPa·m$^{1/2}$ [43], and the bending strength is difficult to exceed 1 GPa [44,45]. Thus, the minimum critical flaw size of existing ceramics can be estimated by $2^2/(3.14\times1000^2)$ m≈1.27 μm, which is slightly larger than the notch tip radius (＜1 μm) introduced by laser method. Ceramics with higher strength are often accompanied by greater fracture toughness, resulting in larger critical flaw size, a_c, and critical notch tip radius, ρ_c (for example, the strength of 2 GPa in ZrO_2-WC nanocomposites corresponds to the K_{Ic} of 9.4 MPa·m$^{1/2}$ and the a_c value of 7.04 μm [46]). Malkin model is also used to assess the minimum ρ_c in ceramics. Taking K_{Ic} of 2 MPa·m$^{1/2}$ [43], σ_Y of 500 MPa [47], E of 541 GPa [48], x^* of 1 μm [36,37], ε_f (R_β) of 0.1% [49] as the limit values, the ρ_c calculated by Eq. (4) is 1.30 μm, which is consistent with the lower threshold value achieved by Eq. (5). Therefore, it could be reasonable to believe that the V-notch produced by laser notching method is sharp enough for all ceramics.

Table 2 Material parameters and mechanical properties of sixteen kinds of ceramics

Materials	Original strength, σ_0 (MPa)	Fracture toughness, K_{Ic} (MPa·m$^{1/2}$)	Three times the average grain size, 3d (μm)	Critical flaw size, a_c (μm)	Critical notch tip radius, ρ_c (μm)	n value
5Y-TZP	913	5.37	~1.5	11.01	~9.41	0.35
2Y-TZP(W)[50]	980	-	-	-	-	0.27
2.5Y-TZP(W)[50]	870	-	-	-	-	0.36
3Y-TZP(W)[50]	780	-	-	-	-	0.32
3Y-TZP[26]	770	4.05	~1.5	8.81	~8.03	0.27
Si_3N_4(J)	664	5.20	~4.5	19.52	~18.55	0.19
Si_3N_4(Y)	283	2.41	~3.6	23.08	~23.43	0.18
Si_3N_4(G)[51]	700	5.40	~12	18.94	17.72	0.33
SiC	305	2.75	~18	25.88	~27.99	0.18
SiC(A)[52]	620	3.70	-	11.34	9.5	0.28

Al_2O_3	307	2.93	~30	28.99	-	-
Al_2O_3(T)[53]	297	3.80	~7.5	52.11	61.40	0.31
Al_2O_3(B)[54]	350	3.10	~30	24.97	45.43	0.43
ZrB_2	456	2.73	~24	14.03	~18.88	0.20
ZrB_2-SiC	740	2.86	~9	4.75	~7.45	0.25
ZrB_2-SiC-G	404	3.62	-	25.56	~36.17	0.19

3.2 Effect of notch depth

The critical stress intensity factor (K_c) values of these typical structural ceramics which are measured by different notch depths are plotted in Fig. 5. It can be seen clearly that the K_c values obtained by SEVNB method are closely correlated with the a/W. In contrast to metals [55], the K_c values increase rapidly when a/W is below a critical value, which might be determined by material itself (i.e., the composition and the microstructure), and then remain constant at the fracture toughness (K_{Ic}). This is consistent with the previous reports that the K_c of shallow notched brittle materials are lower than that for deep notches or large cracks [56].

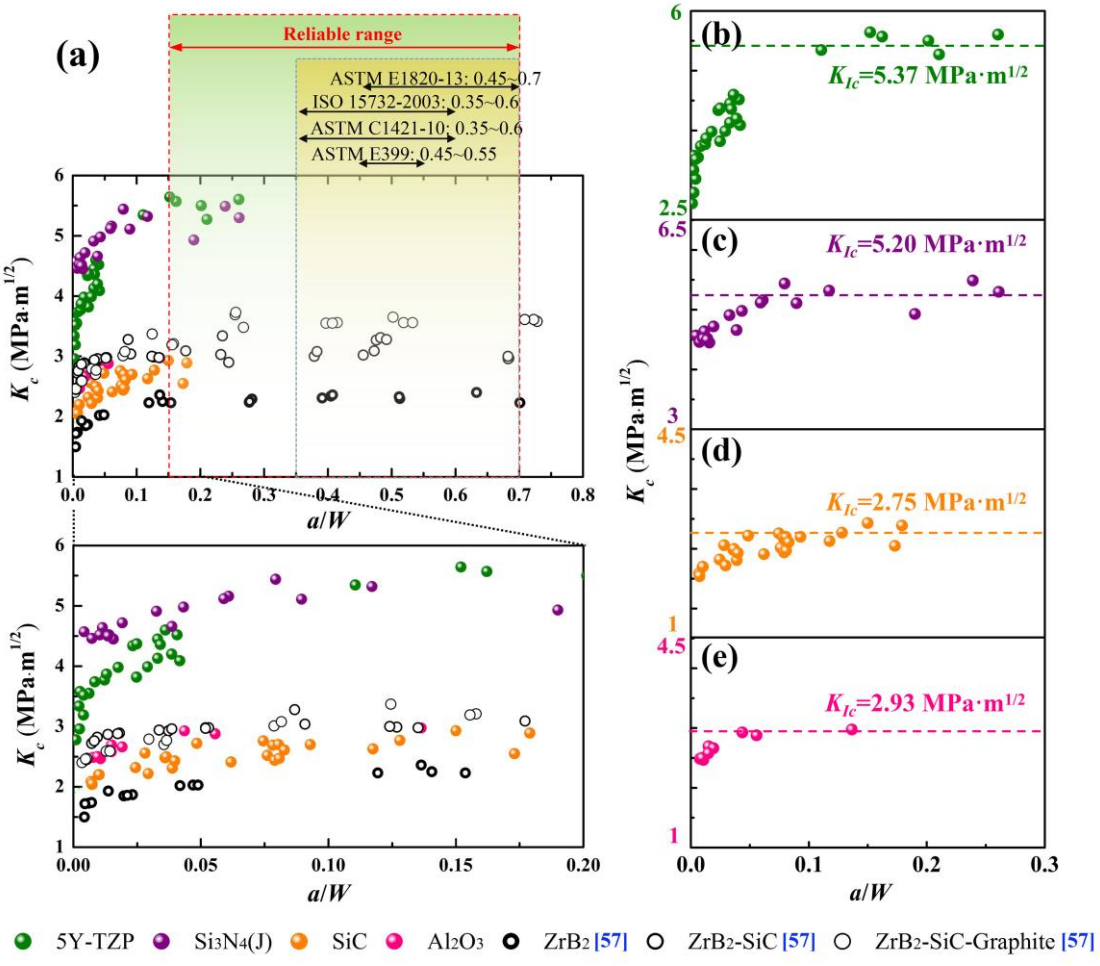

Fig. 5. Fracture toughness of various ceramics with different *a*/*W* measured by laser notched SEVNB method. (a) Our work and the literature report data [57] and (b) 5Y-TZP, (c) Si_3N_4(J), (d) SiC as well as (e) Al_2O_3 ceramics, respectively.

The critical value of *a*/*W* is not more than 0.15 whether it is oxide, nitride, carbide or boride ceramics, and even for ceramic matrix composites containing a large amount of toughened phases (i.e., ZrB_2-20 vol.% SiC-15vol.% Graphite), a relative depth *a*/*W* of 0.25 is also deep enough. Nevertheless, current commonly used test standards prescribe that the *a*/*W* should between 0.35 and 0.7, and the ASTM E399 even requires the relative depth ranging from 0.45 to 0.55. Obviously, the *a*/*W* requirement in current standards seem to be too harsh, this may be because: (*i*) In the past, it is tough to introduce shallow notches (less than 1 mm) into ceramics because the traditional notching methods are difficult to control the notch depth; (*ii*) Making a notch with uniform depth is not trivial, and this will be more difficult especially when the notch is shallow, which in turn leads to large errors in fracture toughness measurements.

By observing the cross-sectional morphology of the ceramic specimens processed by the novel laser notching method, it is found that the notch depth is very uniform (i.e., the depth deviation in 5Y-TZP ceramic does not exceed 8%, and the maximum and minimum depths are 76 μm and 70 μm, respectively, as drawn in Fig. 6), which indicates that the above-mentioned problems could be effectively solved by the laser method. Through adjusting the laser processing parameters, it is clear that the notch bottom is flat at various notch depths (see Fig. 7a-c), while the roughness at the bottom increases slightly with increasing notch depth. In the view of the large enough notch depth, the deviation of the *a*/*W* will not increase significantly and thus the error of the notch depth could be neglected. Furthermore, partially enlarged view indicates that there is no remelting, recrystallization, microcrack or other damages near the notching boundary (as shown in Fig. 7d and e). In the past, Turon-Vinas *et al*. [58,59] observed a narrow microcracked region (damage zone) in front of the notch tip in ceramics after laser processing. Subsequently, Zhao *et al*. [31] revealed that the

damage zone in front of V-notch tip was just superfical and could be easily removed by polishing. In addition, based on the fracture analysis and comparison of the measured values before and after acidic pickling (hydrofluoric acid) as well as annealing for Si_3N_4 [31], 3Y-TZP [32], Al_2O_3 [32,33] and 8Y-FSZ [32] ceramics, they further proved that the effect of the physical changes of the material (such as remelting and recrystallization) and the residual thermal stress on the fracture toughness measurement results could be ignored. Our previous researches also excluded the influence of laser processing residual stress [11,26,57]. In view of this, we believe that the appropriate nanosecond laser processing has only a minimal/negligible effect on the microstructure of the material, thus achieving the true fracture toughness in ceramics during SEVNB test.

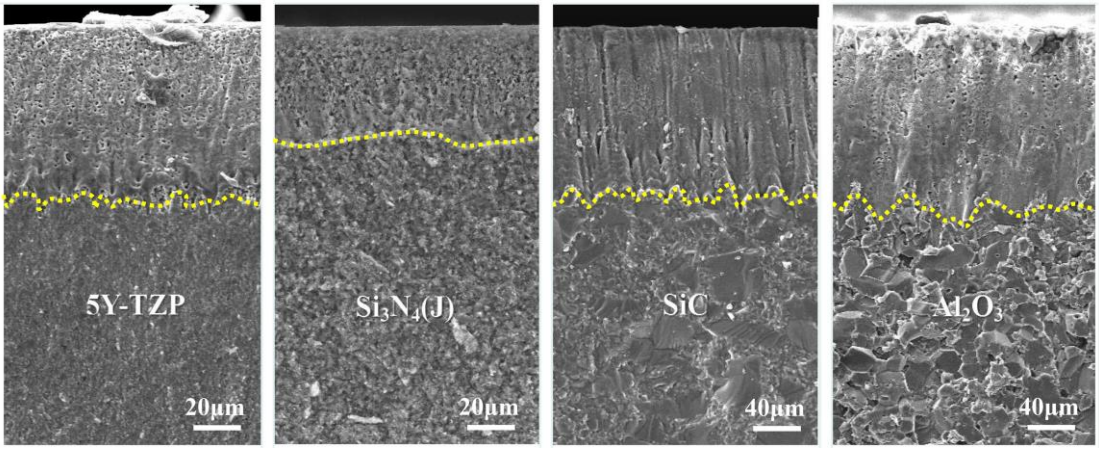

Fig. 6. Fracture surfaces of test ceramics notched by nanosecond laser.

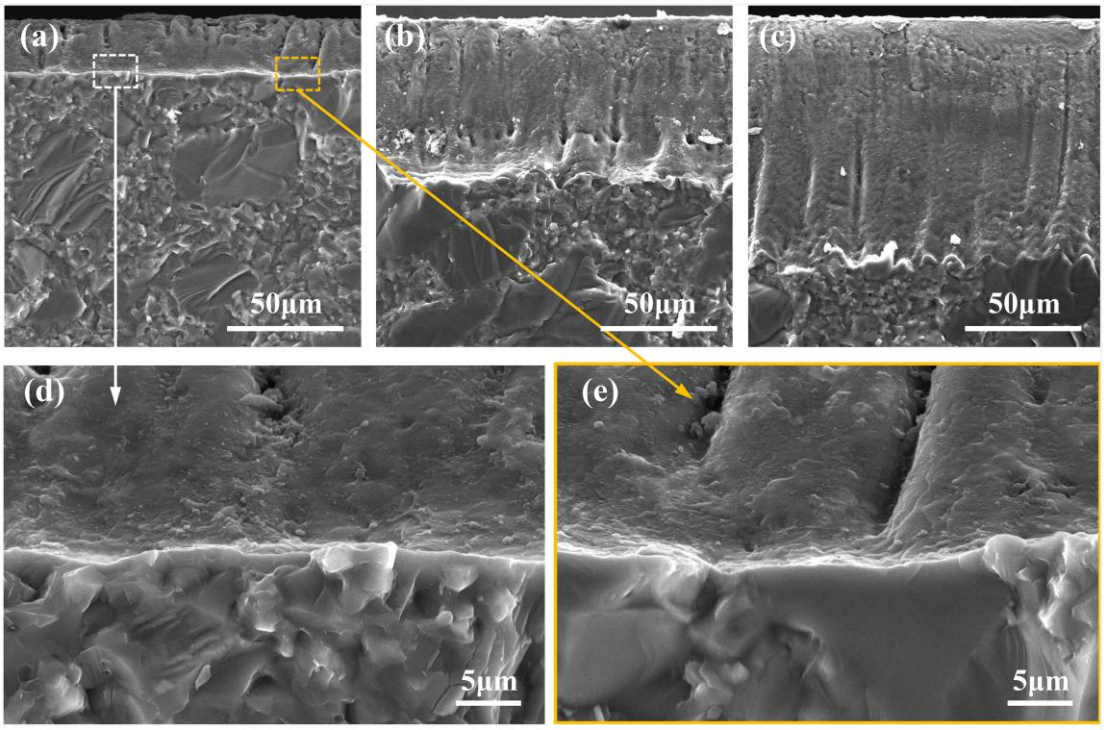

Fig. 7. Fracture surfaces of SiC ceramic with various notch depth.

Since the fracture toughness is calculated by fracture strength (σ) as well as relative depth (a/W) values, if the relationship between σ and notch depth (a) or a/W are well established, the fracture toughness measurement could be guided more effectively. As depicted in Fig. 8, the experimental data of σ vs a was analyzed in double logarithmic coordinates based on the linear elastic fracture mechanics (LEFM). The strength values of these ceramics decrease linearly with the notch depth, which is close to those previous studies of typical ceramics containing indentation [60,61] and femtosecond laser processing pore-like flaws [62]. Linear regression was then applied, and it is exciting that the slope value m proposed by LEFM is not constant at -0.5 but depending on material itself to a large extent (m is -0.383, -0.372, -0.381 and -0.395 for 5Y-TZP, Si_3N_4(J), SiC and Al_2O_3 ceramics, respectively).

In theory, a flaw-free sample should be able to sustain a mechanical stress near the theoretical strength (σ_{th}) of solid. Thus, linear extrapolations to very shallow notch sizes (atomic scale, ~3 Å in Ref. [63]) were put into effect, and the estimated ultimate strength (σ_{max}) is about 20.9, 30.8, 17.4 and 22.0 GPa for 5Y-TZP, Si_3N_4(J), SiC and Al_2O_3 ceramics, respectively. Rely on the relationship between theoretical strength and elastic modulus (σ_{th} is in the order of ~E/10 to E/30 in Ref. [64]), and by applying

the elastic modulus values as reported in Refs. [65-68], we can estimate that the σ_{th} is 7.7-23.0, 11.2-33.5, 11.7-35.0 and 13.3-40.0 GPa for 5Y-TZP, Si_3N_4(J), SiC and Al_2O_3 ceramics, respectively. It is exciting that the theoretical σ_{max} values for these four typical structural ceramics obtained by linear extrapolation are in good agreement with the σ_{th} values. Similar phenomenon has also been revealed in boride ceramics [57]. In light of the above-mentioned exponential relationship between fracture strength (σ) and notch depth (a), it can be used not only to predict the theoretical strength (σ_{th}) of ceramics, but also to deduce the σ value at different a when the σ_{th} is known (such as first-principles calculations [69]), so as to obtain the relationship between critical stress intensity factor (K_c) and a according to the fracture toughness calculation formula.

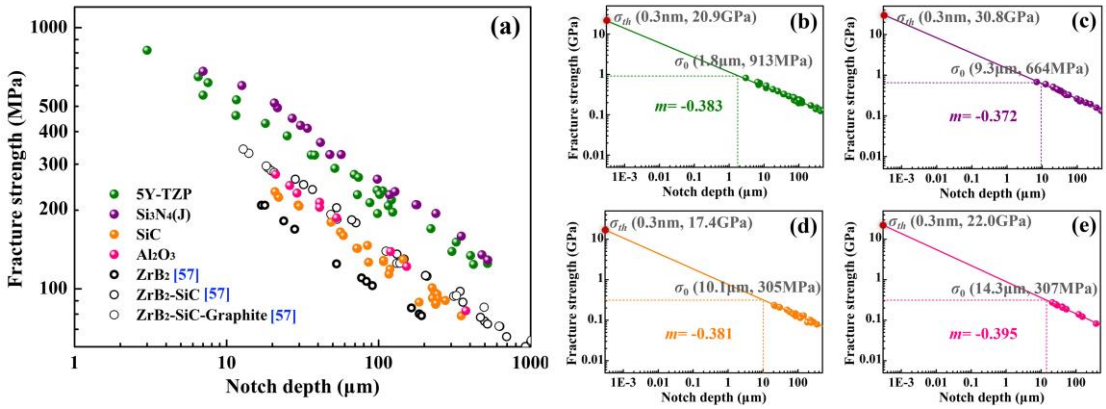

Fig. 8. Relationship between fracture strength and notch depth for various ceramics in double logarithmic coordinates. (a) Our work and the literature report data [57] and (b) 5Y-TZP, (c) Si_3N_4(J), (d) SiC as well as (e) Al_2O_3 ceramics, respectively.

3.3 Effect of equivalent notch angle

To the author's best knowledge, the laser notch depth is often less than 500 μm (even laborious to achieve 100 μm via the femtosecond laser as reported in Refs. [28,58]), which is far less than the depth of at least 1 mm that recommended in test standards. Thus, the laser notching approaches should be utilized in combination with the traditional U-groove notching methods, resulting in an equivalent notch angle (θ) which is determined by the laser notch depth and the U-groove width. Here, the measured values of fracture toughness (K_{Ic}) at different θ are plotted in Fig. 9. It is obvious that when θ is below about 60°, the K_{Ic} value stays almost constant at the

intrinsic value (or name it the actual value), and then increases gradually as the θ increases. Similar to those reported by GÓMEZ [70] for some brittle or quasi-brittle materials with good processability, the approximate expression of the measured K_{Ic} value can be expressed as:

$$K_{Ic,measured}= [1+0.038393(\pi\theta/180)^2-0.027857(\pi\theta/180)^3+0.024207(\pi\theta/180)^4]\cdot K_{Ic,actual} \quad (6)$$

where $K_{Ic,actual}$ is obtained by sharp V-notches, θ ranges from 0° to 160°.

It can be seen that the experimental results agree well with the predicted values (solid lines in Fig. 9). From the predicted curves, it is clear that the $K_{Ic,measured}$ could be regarded as being accurate when the θ is less than 60°. However, there is an inevitable deviation between the center lines of the laser V-notch and the U-groove during notching process, which in turn leads to the measurement errors of K_{Ic}. For instance, the θ of the two typical notches of 5Y-TZP ceramic are basically the same as 65° and 67°, see Fig. 9a, respectively, wherein the former is almost symmetrical along the notch centerline and the error between the experimental as well as the prediction results is less than 3%, while a 10% mistake is exhibited in the latter one, which may owing to the 0.09 mm deviation between the laser V-notch and the U-groove. It is not trivial to control the position of the V-notch on most commercial lasers equipment, especially on the order of 0.1mm, thus, in order to accurately measure the fracture toughness, the θ should be small enough, which requires reducing the U-groove width and increasing V-notch depth as much as possible. At present, the θ recommended by ASTM E1820-13 [4] and ISO 23146-2012 [8] are 60° and 30°, respectively, and the latter standard would be more conducive to obtaining the accurate fracture toughness value.

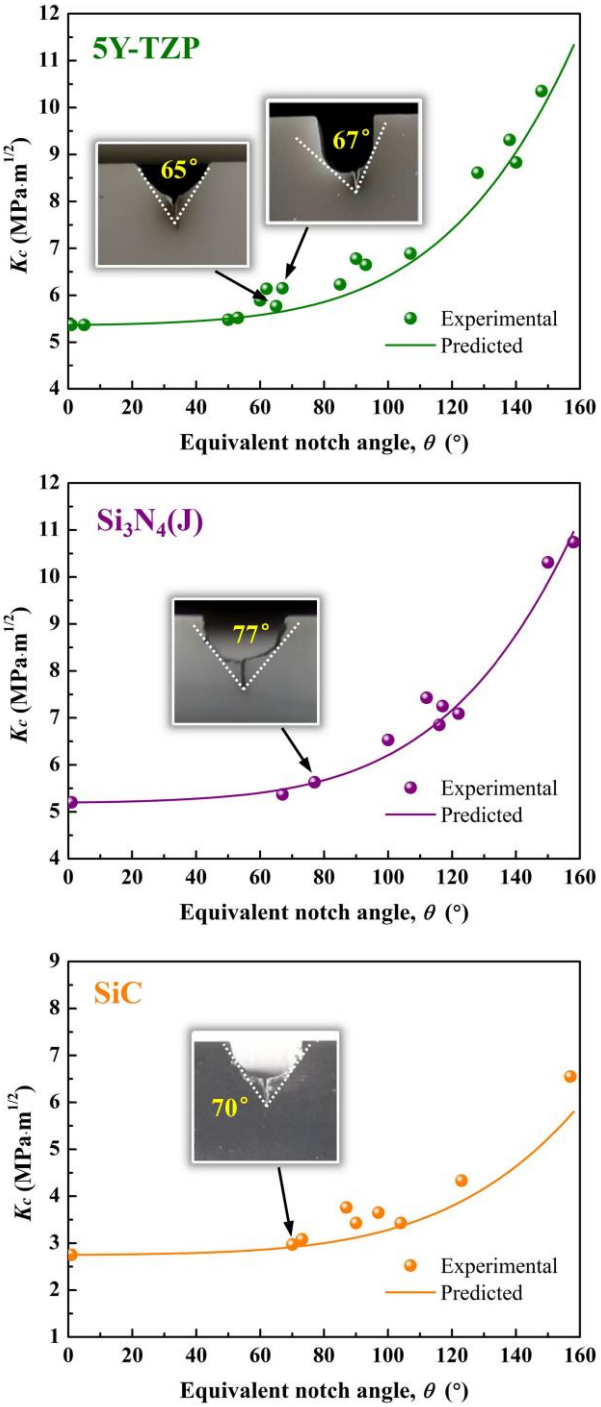

Fig. 9. Experimental and predicted values of K_c in 5Y-TZP, Si_3N_4(J) and SiC samples with various equivalent notch angles (θ).

3.4 Effect of sample size

In order to meet the current standards, it is unavoidable to perform a traditional notching process before laser notching, which restricts the important advantages of laser method as simple and efficient. If a thinner specimen is used, only the laser

notching approach is required to achieve a sharp V-notch with satisfactory sharpness (ρ＜1 μm), sufficient relative notch depth (a/W＞0.15) and ideal equivalent notch angle (θ＜10°). Here, smaller and thinner specimens were used for the SEVNB test, and the test results were compared with that of the current standards of ASTM E1820-13 [4] and GB/T 23806 [9], as plotted in Fig. 10. The small-sized sample test results of the five kinds of ceramics are in good agreement with the results of the standard samples (error does not exceed ±5%), indicating that the reduction in sample size will not affect the test accuracy. Miyazaki *et al*. [71] have also measured the fracture toughness of typical ceramics (including Al_2O_3, AlN and Si_3N_4) thin plates with thickness of 0.32 mm and 0.64 mm by means of several testing institutions, and a similar conclusion is given that the sample thickness has no effect on the measured value.

Therefore, using small and thin SEVNB samples could give full play to the advantages of laser notching method without reducing the test accuracy, especially in the case of limited billet or substrate in the development of new materials, it can replace the indentation method, which often comes at the cost of losing part of the test precision and reliability.

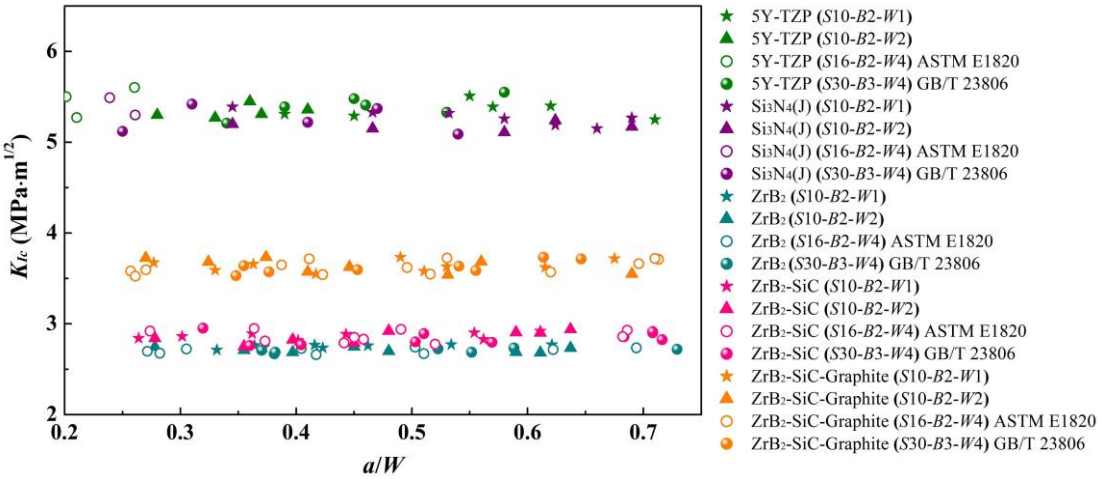

Fig. 10. Experiment results of sample size effect on the measurement of fracture toughness in 5Y-TZP, Si_3N_4(J), ZrB_2, ZrB_2-SiC and ZrB_2-SiC-Graphite ceramics.

4. Conclusions

In order to promote the standardization of laser notching method, in this work, the effects of laser notch tip radius (ρ), notch depth (a), equivalent notch angle (θ) and

sample size on the measured value of fracture toughness (K_{Ic}) were systematically studied. Following are the main conclusions:

(1) The laser notching method is a very versatile method, which seems to be applicable to all ceramic materials (including oxide, nitride, carbide and boride ceramics). The V-notch tip radius prepared by laser method can be easily lower than 1 μm, which is one order of magnitude lower than the traditional methods.

(2) In double logarithmic coordinates, the K_{Ic} value almost increases linearly when the ρ exceeds the critical notch tip radius (ρ_c), namely, the K_{Ic} value is proportional to ρ^n, where n is normally in the range of 0.1-0.5 depending on the material itself. For fine-grained ceramics with submicron grain size (3Y-TZP and 5Y-TZP), the critical tip radius (>5 μm) is still far from reaching the limit of laser notching method.

(3) For monolithic ceramics, the K_{Ic} value will reach a plateau when the relative notch depth (a/W) exceeds 0.15, which is much lower than 0.35 that is recommended in existing standards.

(4) The relationship between the K_{Ic} value and θ in ceramics follows well with the GÓMEZ criterion, that is, the θ should be at least less than 60° in order to avoid overestimation of K_{Ic}. Considering that it is challenging to perfectly overlap the V-notch and the U-groove during processing, which inevitably leads to poor consistency and overestimation of measured values of K_{Ic}, it could be more reasonable to refer to the requirement given by ISO 23146-2012 (θ＜30°).

(5) The SEVNB technique combined with laser notching method can be applicable to various ceramic small and thin plates with thickness from 1 to 2 mm.

Accordingly, it is safe to conclude that fracture toughness can be accurately measured by using small specimens with the thickness of less than 2mm in conjunction with the laser direct-notching method. In this case, the outstanding advantages of the laser method, such as fast, good efficiency, low cost as well as low material consumption, can be fully exploited.

Acknowledgement

Financial support was provided by the Natural Science Foundation of Jiangsu

Province (No. BK20201040), the Surface Projects of Natural Science Research in Jiangsu Province (No. 19KJB430021), the Special Talent Introduction for "Double Innovation Plan" in 2019, the University Research Foundation of Nanjing Institute of Technology (No. YKJ201806), the National Natural Science Foundation of China (No. 52002108), the Natural Science Foundation of Hebei Province (No. E2020202002) and the Outstanding Scientific and Technological Innovation Team in Colleges and Universities of Jiangsu Province.

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Figures

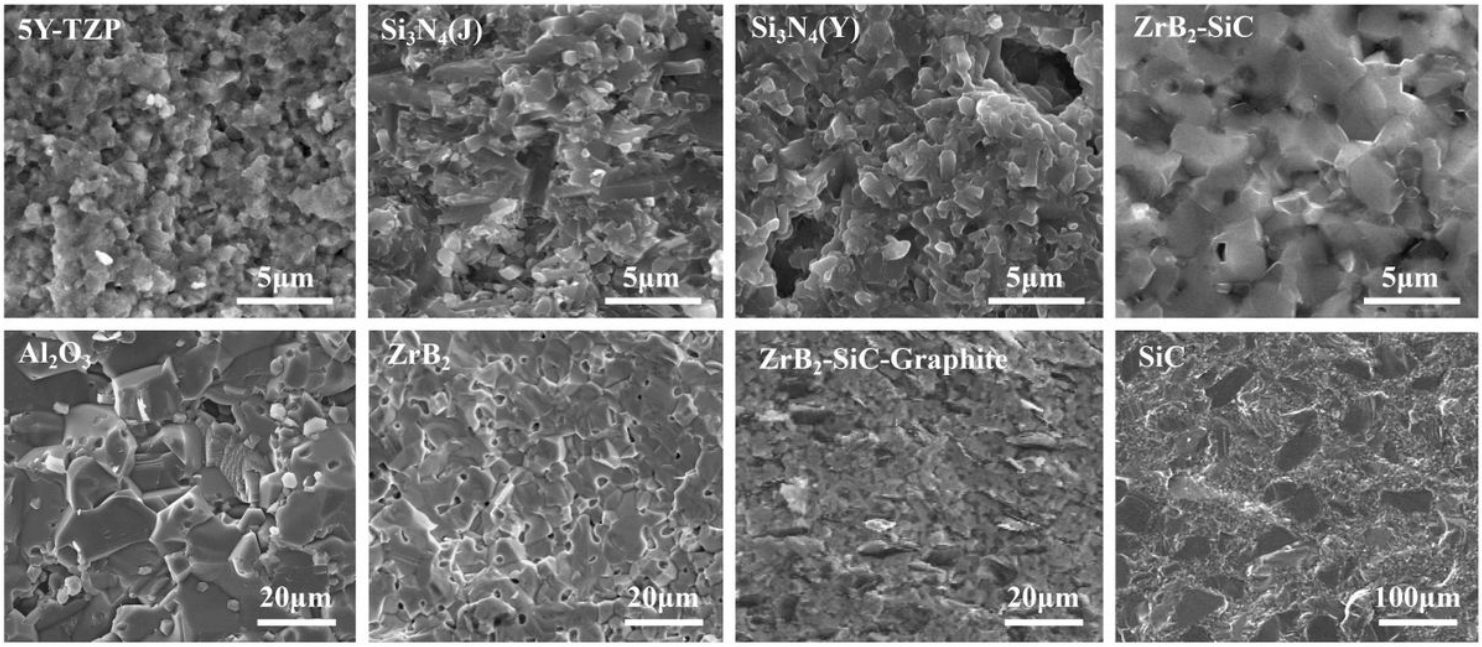

Figure 1

SEM images of fracture surfaces of test samples.

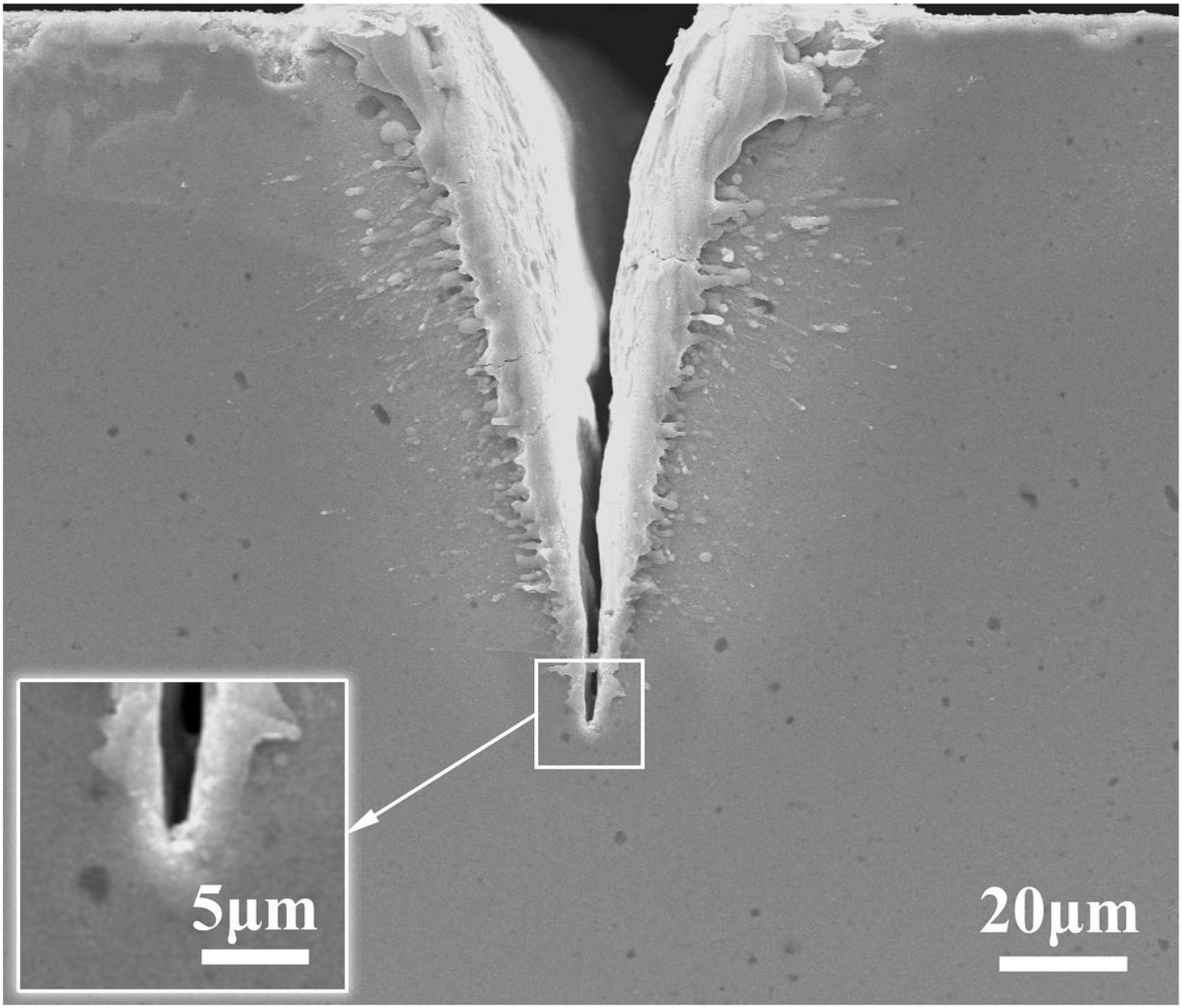

Figure 2

SEM image of 5Y-TZP ceramic bar with an ultra-sharp V-notch. The enlarged view shows that the V-notch tip radius is ~0.7 μm and no microcrack is found near the V-notch root.

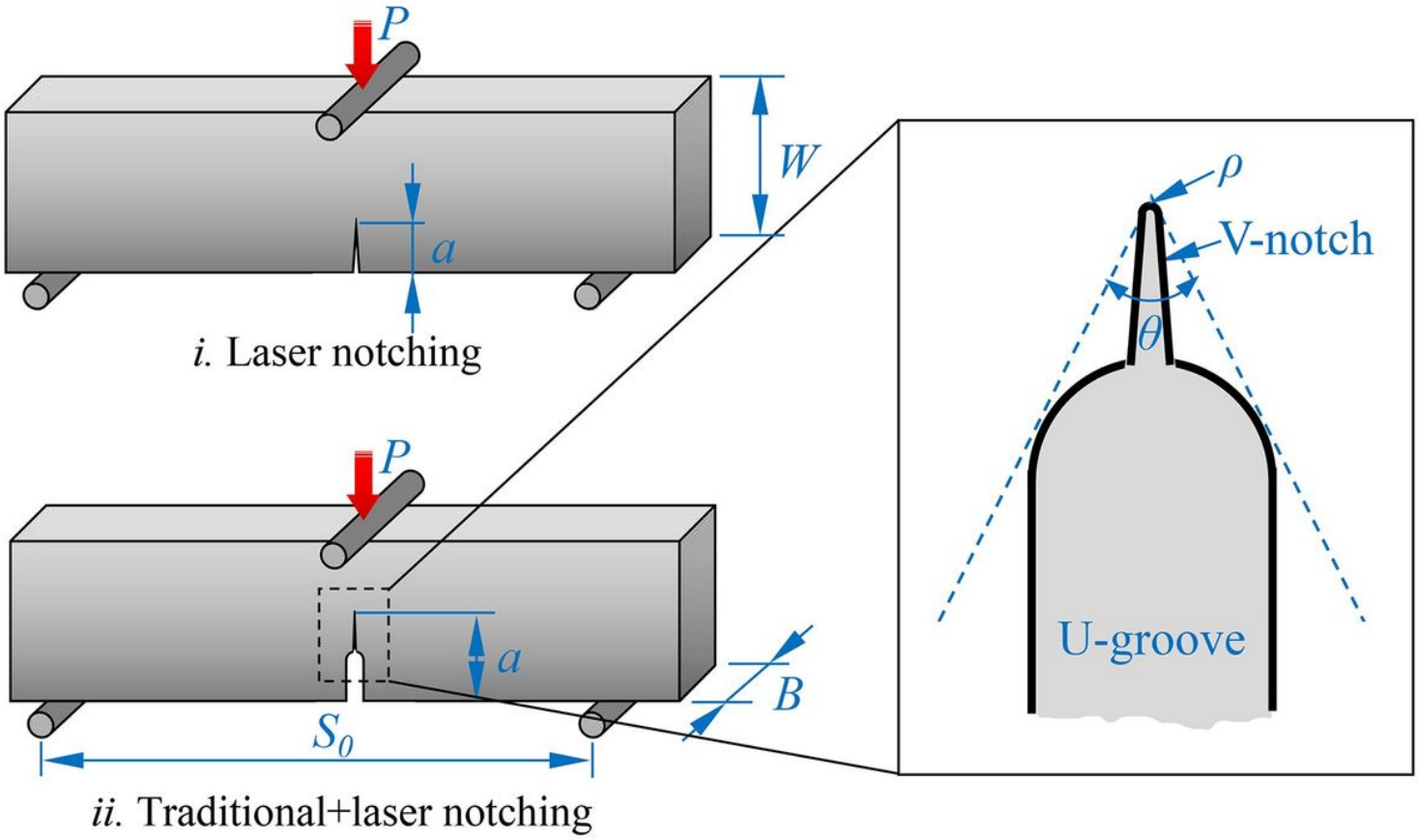

Figure 3

Schematic of the SEVNB test specimens with sharp notches.

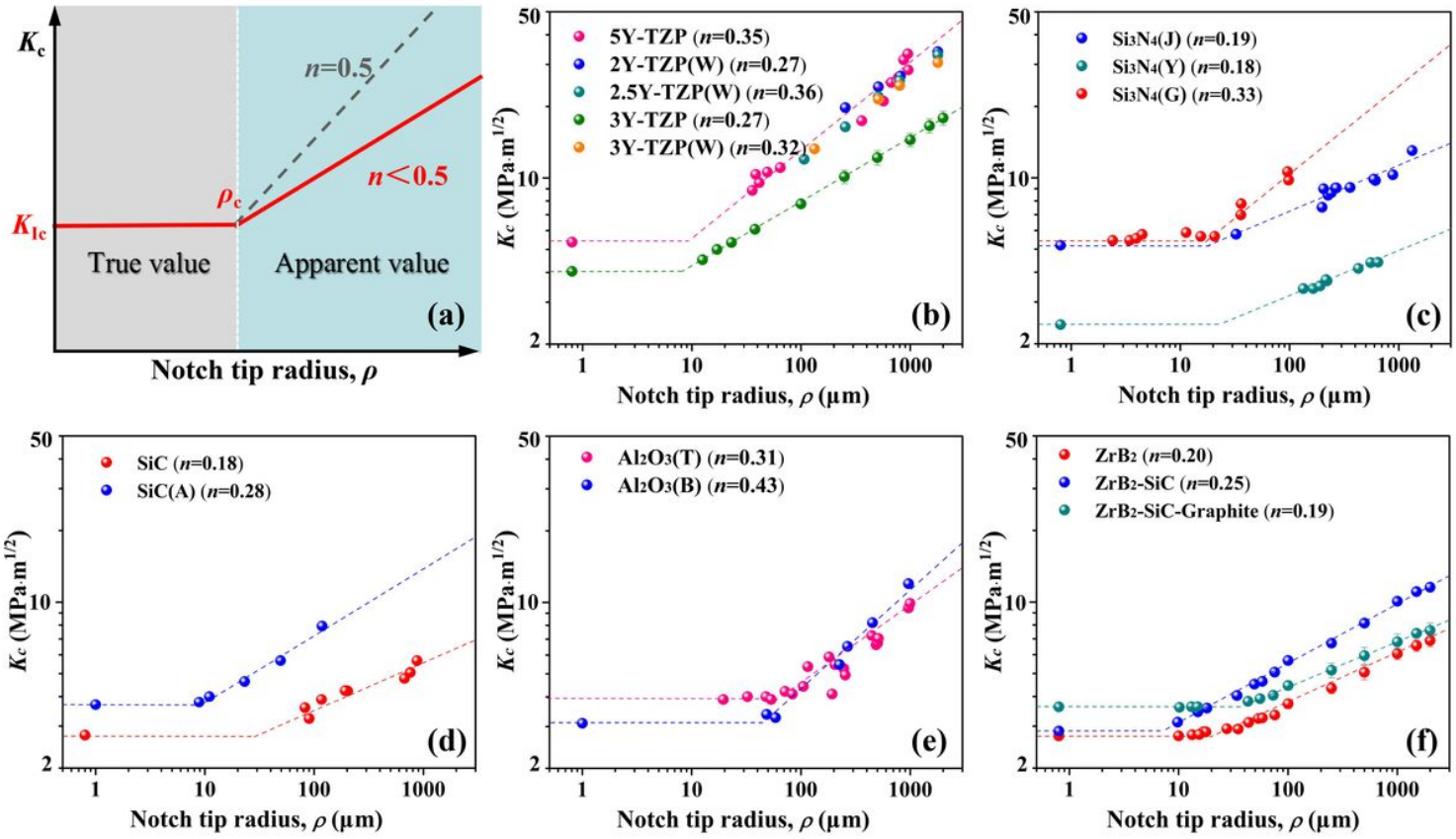

Figure 4

Variation of the critical stress intensity factor (Kc) with notch tip radius (ρ). (a) Schematic diagram and test results of (b) yttria-stabilized zirconia, (c) silicon nitride, (d) silicon carbide, (e) alumina as well as (f) ZrB2-based ceramics in double logarithmic coordinates.

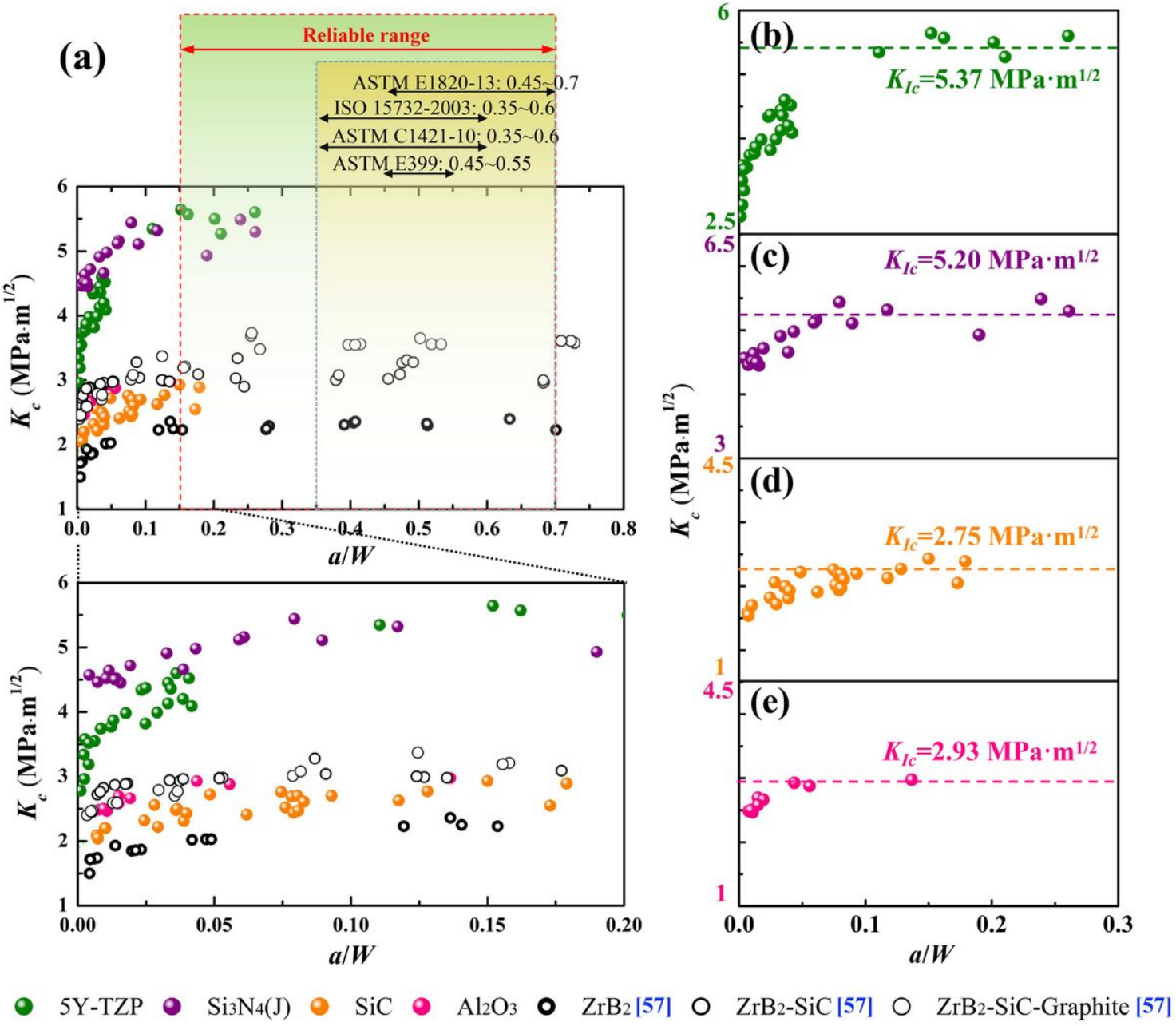

Figure 5

Fracture toughness of various ceramics with different a/W measured by laser notched SEVNB method. (a) Our work and the literature report data [57] and (b) 5Y-TZP, (c) Si3N4(J), (d) SiC as well as (e) Al2O3 ceramics, respectively.

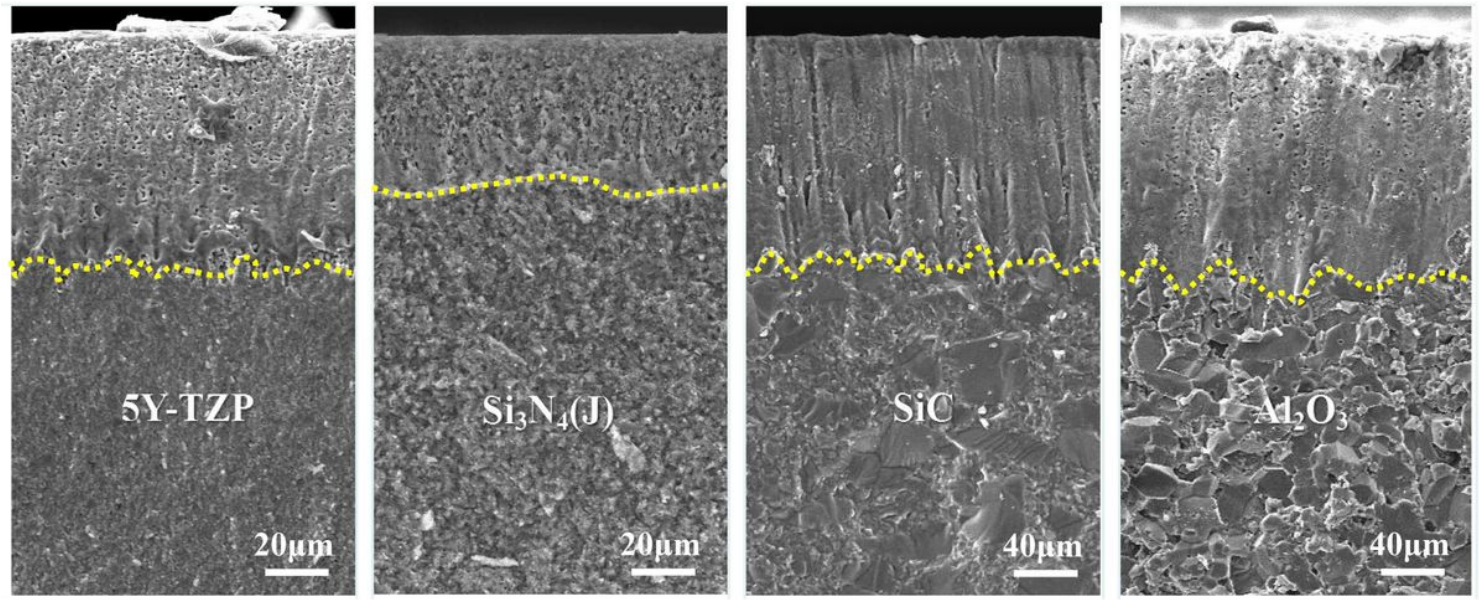

Figure 6

Fracture surfaces of test ceramics notched by nanosecond laser.

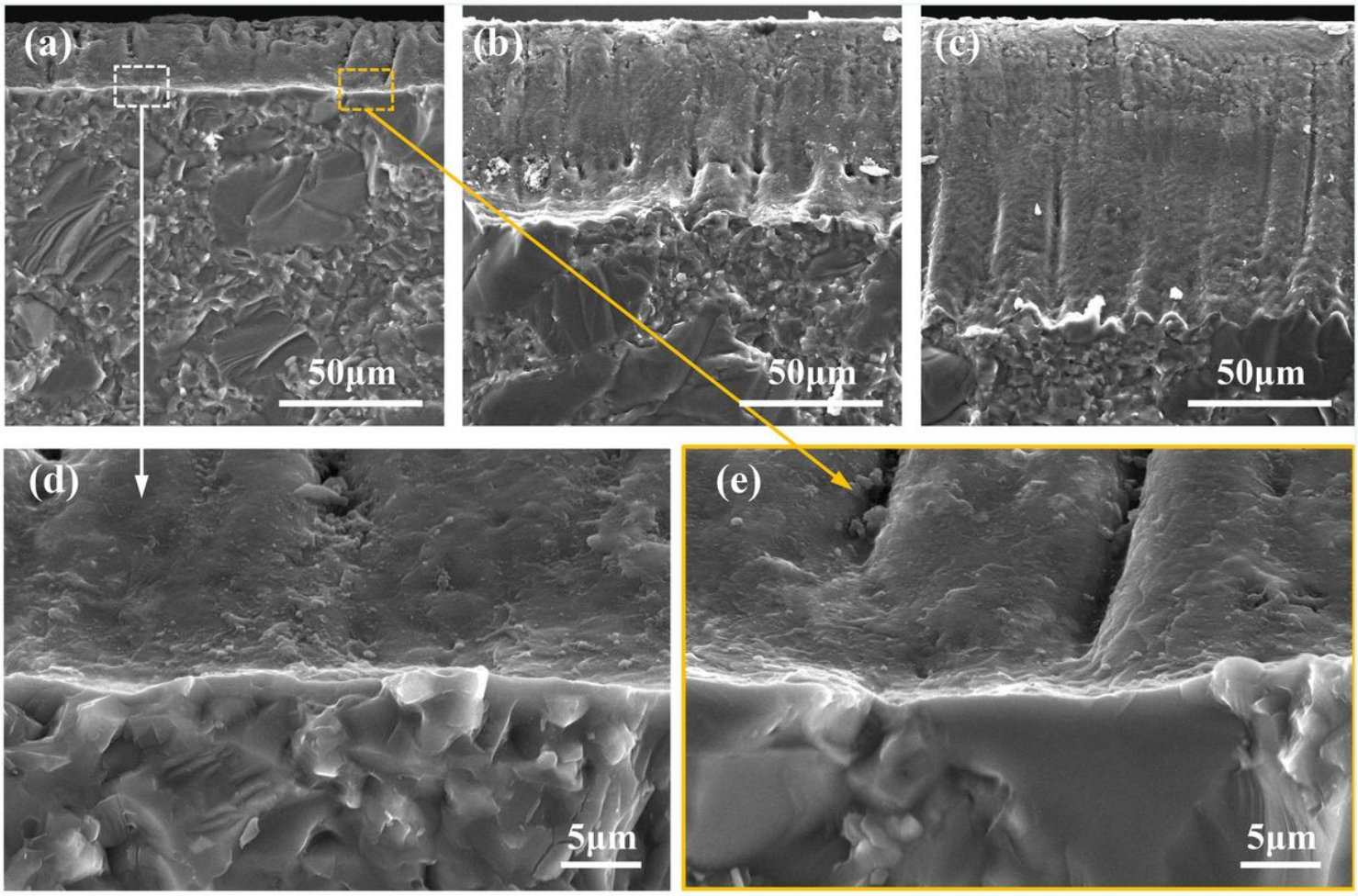

Figure 7

Fracture surfaces of SiC ceramic with various notch depth.

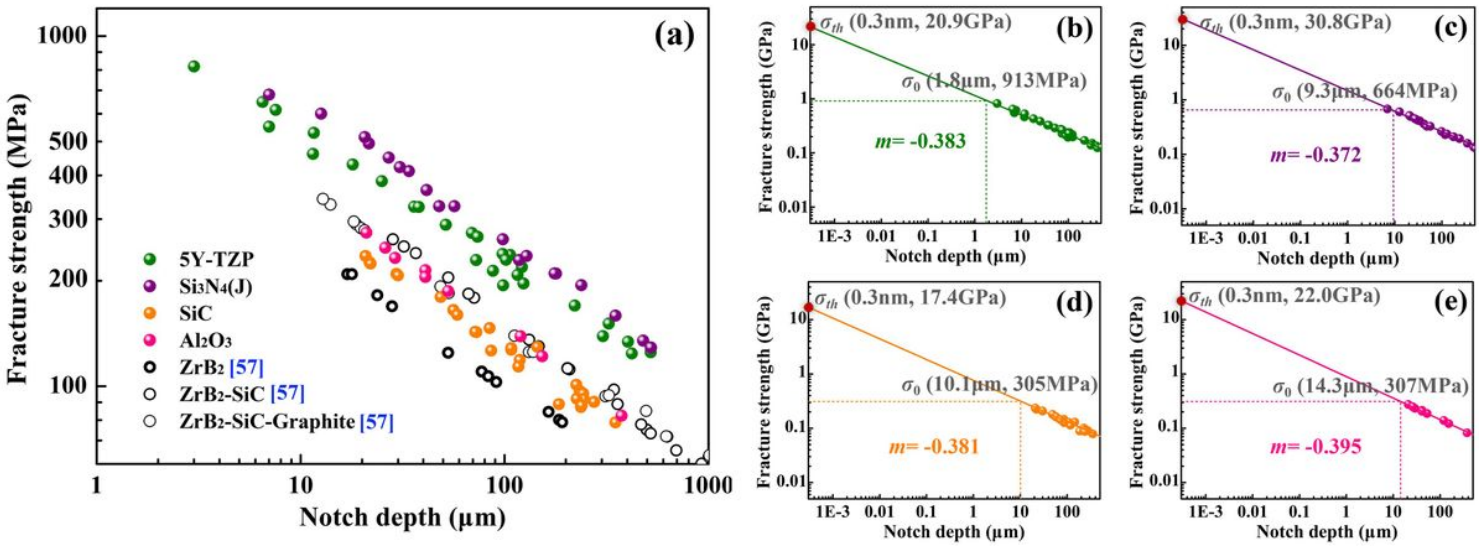

Figure 8

Relationship between fracture strength and notch depth for various ceramics in double logarithmic coordinates. (a) Our work and the literature report data [57] and (b) 5Y-TZP, (c) Si3N4(J), (d) SiC as well as (e) Al2O3 ceramics, respectively.

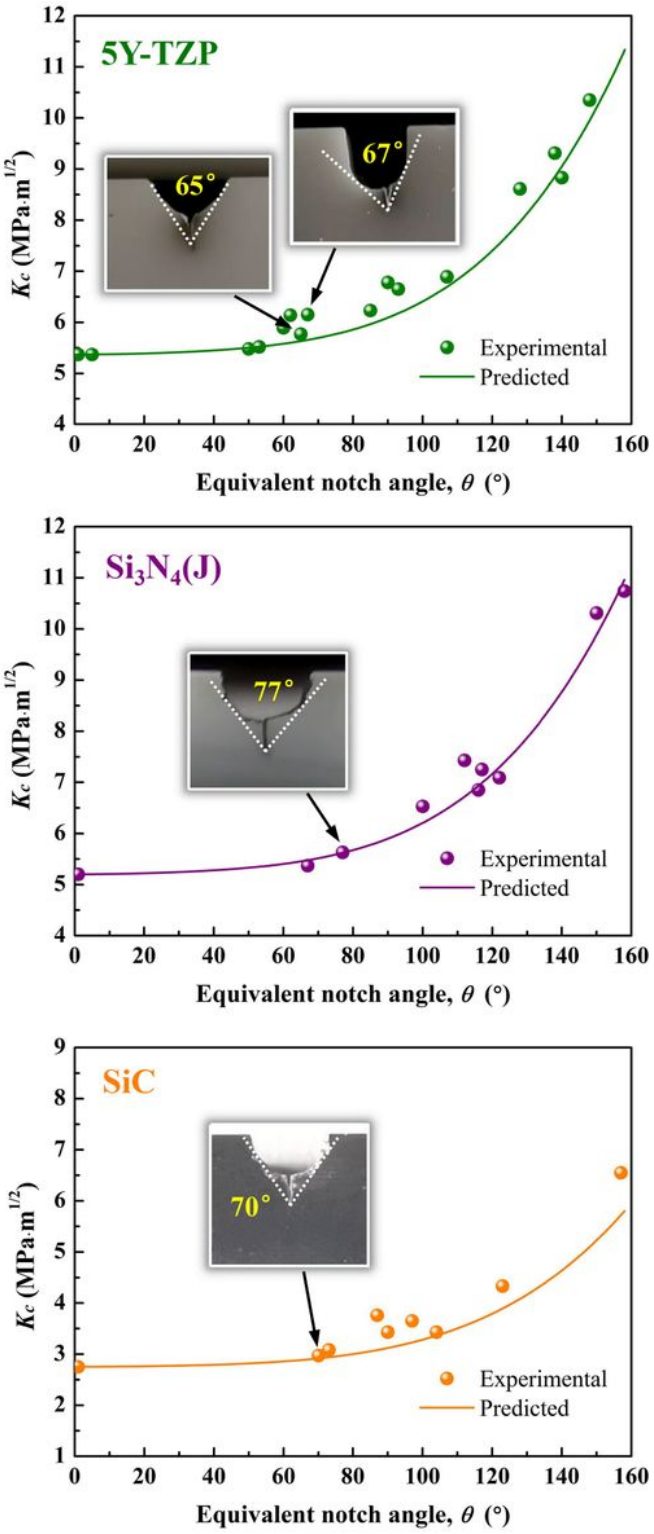

Figure 9

Experimental and predicted values of Kc in 5Y-TZP, Si3N4(J) and SiC samples with various equivalent notch angles (θ).

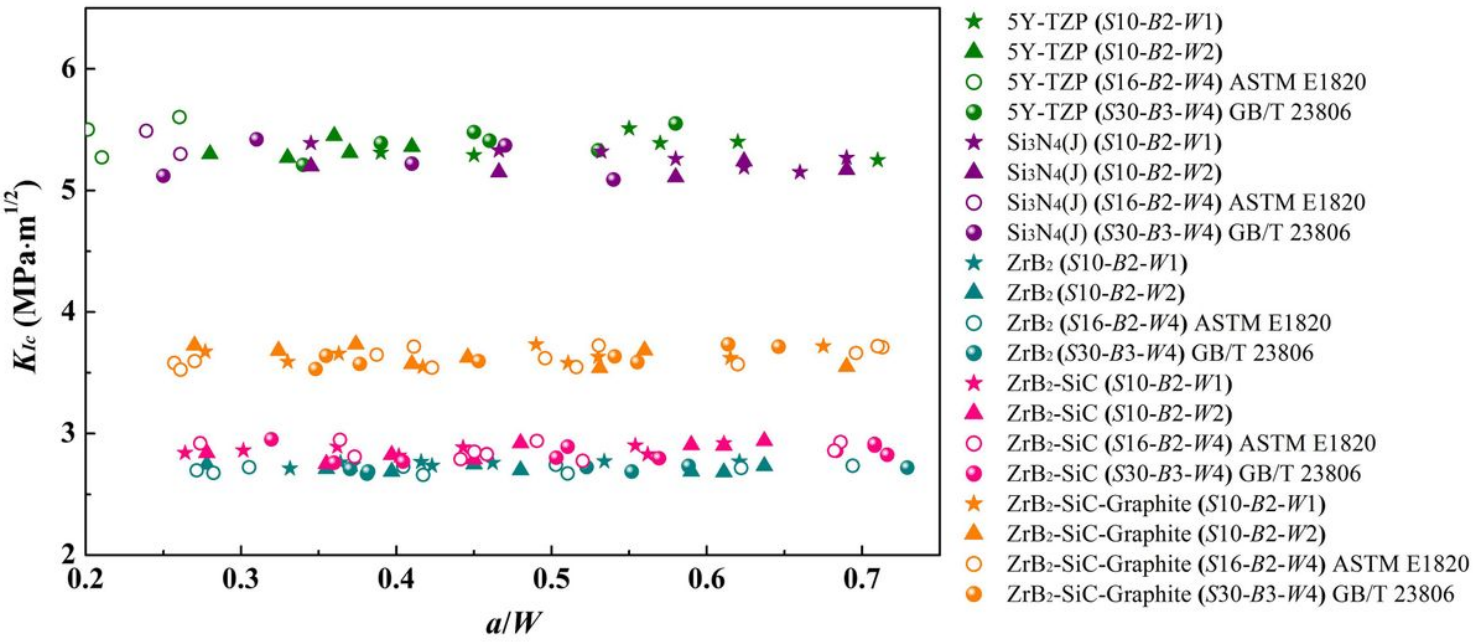

Figure 10

Experiment results of sample size effect on the measurement of fracture toughness in 5Y-TZP, Si3N4(J), ZrB2, ZrB2-SiC and ZrB2-SiC-Graphite ceramics.