Journal of the European Ceramic Society 37 (2017) 4207–4212

Contents lists available at www.sciencedirect.com

Journal of the European Ceramic Society

journal homepage: www.elsevier.com/locate/jeurceramsoc

Feature article

Accurate measurement of fracture toughness in structural ceramics

Anzhe Wang [a], Ping Hu [a,b,c,*], Xinghong Zhang [a,**], Wenbo Han [a], Guiqing Chen [a], Jiecai Han [a,b]

[a] Science and Technology on Advanced Composites in Special Environment Laboratory, Harbin Institute of Technology, Harbin, 150080, PR China
[b] State Key Laboratory of Advanced Welding and Joining, Harbin Institute of Technology, Harbin, 150001, PR China
[c] Key Laboratory of Materials Physics, Institute of Solid State Physics, Chinese Academy of Sciences, Hefei, 230031, PR China

ARTICLE INFO

Article history:
Received 24 February 2017
Received in revised form 10 May 2017
Accepted 13 May 2017
Available online 27 May 2017

Keywords:
Fracture toughness
Structural ceramics
Laser
V-notch
Notch tip radius

ABSTRACT

The measured values of fracture toughness for ceramics are closely correlated with the sharpness of notch tips, which in turn influences the accurate measurement of fracture toughness. Here, typical structural ceramics, i.e., 3 mol% yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP), ZrB_2, ZrB_2-SiC and ZrB_2-SiC-Grapite, were used for the measurement of fracture toughness, and the effect of notch tip radius on the fracture toughness values of these typical structural ceramics was investigated. Ultra-sharp notches with a tip radius less than 1 μm can be fabricated by laser, lower than the critical notch tip radius in ceramics below which the fracture toughness value almost remains constant, and improved accuracy and consistency of fracture toughness measurement can be obtained by this method compared with traditional method.

1. Introduction

In the past decades, there have been considerable efforts directed at developing and evaluating meaningful fracture toughness test methods for structural ceramics [1]. Unfortunately, although a variety of test methods, such as chevron notch (CN) [2,3], surface crack in flexure (SCF) [4,5], double cantilever beam (DCB) [6], indentation fracture (IF) [7,8], single-edge notched beam (SENB) [9], single-edge precracked beam (SEPB) [10,11] and single-edge V-notched beam (SEVNB) [12] methods have been proposed, no consensus has been established yet to evaluate the fracture toughness, K_{Ic}, of structural ceramics, because the values measured by different methods are randomly scattered to a great extent [13–15].

The CN method is generally preferred over the SCF method because it produces more consistent results if the notches are properly fabricated. However, the precision fixtures are expensive to fabricate and the saw cutting process is time consuming [16]. A disadvantage of the DCB specimen is that grooves must be used to guide the crack extension along the center line of the specimen and the crack interactions with the groove produce some difficulties in determining K_{Ic} [17]. In the case of IF method, large uncertainties of the test results always arise, which are derived from a number of factors including subjectivity of crack length measurement, variability of indentation crack shapes ranging from half-penny to Palmqvist, susceptibility of residual stress in the prepared surface, and the results may be indentation force and calculation formula dependent [7,8,18].

SENB method is most commonly used in fracture toughness measurement, but the introduction of a sharp crack or notch still remains a challenge [19,20]. SEPB method overcomes the drawback of SENB method, but reliable introduction of a precrack into the ceramic sample is difficult and the crack depth is uncontrollable [11,20–23]. In order to overcome the disadvantages of SENB and SEPB methods, SEVNB method is developed which requires very little specialized apparatus and seems to be more reproducible both within and between laboratories [24]. The V-shaped notch is often processed using a razor blade sprinkled with diamond paste, and the relative basis works were conducted by Awaji [12], Nishida [25] and Fischer *et al.* [26–28]. In particular, Kübler [29] used a razor sprinkled with 1 μm diamond paste to achieve ultra-sharp V-notches on fine-grained 3 mol% yttria-stabilised tetragonal zirconia samples. The K_{Ic} of this material with a notch tip radius, $\rho = 16$ μm, is 5.48 ± 0.30 MPa·m$^{1/2}$, while it decreases to 4.69 ± 0.09 MPa·m$^{1/2}$ when $\rho < 3$ μm. However, fabricating an ultra-sharp V-notch with ρ less than 2 μm by razor blades remains a difficult task. It is not only a time consuming procedure, but also uncontrollable in

* Corresponding author at: Science and Technology on Advanced Composites in Special Environment Laboratory, Harbin Institute of Technology, Harbin, 150080, PR China.
** Corresponding author.
E-mail addresses: huping@hit.edu.cn (P. Hu), zhangxh@hit.edu.cn (X. Zhang).

http://dx.doi.org/10.1016/j.jeurceramsoc.2017.05.027
0955-2219/

keeping same radius on both sides of the V-notch tip. In order to solve these problems and obtain an ultra-sharp V-notch on structural ceramic samples quickly, controllably and reproducibly, laser micromachining technique has been attracted interest due to its great superiority in efficient and high-precision processing [30–34].

The aim of this study was to introduce a convenient, reliable and inexpensive method to obtain an ultra-sharp V-notch with the tip radius less than 1 μm using laser-assisted-machining, which could be used for the accurate measurement of the fracture toughness of structural ceramics, i.e., 3Y-TZP, ZrB_2, ZrB_2-SiC and ZrB_2-SiC-Grapite. The fracture toughness value was compared with the result from bridge indentation (BI) method, which has a real crack and the measured value is considered as the intrinsic value, to evaluate the applicability and effectiveness of the proposed laser notching method. The effect of notch tip radius on the fracture toughness was also investigated.

2. Experimental procedures

2.1. Material and test samples

3Y-TZP ceramic used for fracture toughness measurement was provided by Shenzhen Jinlongda Ceramic Technology CO., LTD. Commercial ZrB_2 (mean particle size is 2 μm, purity >99.5%, Northwest Institute for Non-ferrous Metal Research, China), SiC (mean particle size is 0.5 μm, purity >99%, Aladdin Industrial Corporation, China) and graphite flake (mean diameter and thickness is 15 μm and 2 μm, respectively, purity >99.0%, Qing-dao 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 with WC balls and ethanol as milling media at a speed of 200 rpm. After mixing, the solvent was removed at 70 °C by rotary evaporation to minimize segregation during drying. Uniformly mixed ZrB_2, ZrB_2-SiC and ZrB_2-SiC-G powder mixtures were hot pressed in a BN coated graphite die at 1950 °C for 1.5 h under a uniaxial load of 30 MPa in Ar atmosphere, the heating rate used was ~15 °C/min. After sintering, the furnace was cooled at ~20 °C/min to room temperature under flowing Ar. 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.

2.2. Traditional notching methods

U-grooves with different notch tip radius on the 2 mm × 22 mm surfaces in conductive (ZrB_2, ZrB_2-SiC and ZrB_2-SiC-G) and non-conductive (3Y-TZP) ceramic samples were fabricated using wire cutting machine and diamond wheels, respectively, and the ratio of notch depth to sample height (a/W) was controlled within 0.45–0.55. To get sharper notches with different tip radius, V-notches were polished along the prefabricated U-grooves using razor blades sprinkled with the 1–20 μm diamond pastes.

2.3. Laser notching method

Laser (Proton Laser Applications, Spain) with a power of 1–100 W and a scan speed of 10–50 mm/s was used to fabricate V-notches on the center of the U-groove roots of the test bars. The laser notching parameters, i.e., wavelength, pulse width, repetition rate, power and scan speed were selected by making and assessing practice notches on ceramics. These tests were performed with preselected parameters that would optimize notch quality, and the optimized result of laser notching on the ceramics is presented in Fig. 1. By adjusting the notching times, notches with different depths (generally less than 500 μm) can be fabricated, and for the particular deep ones, laser notching method can be used in combination with the traditional methods.

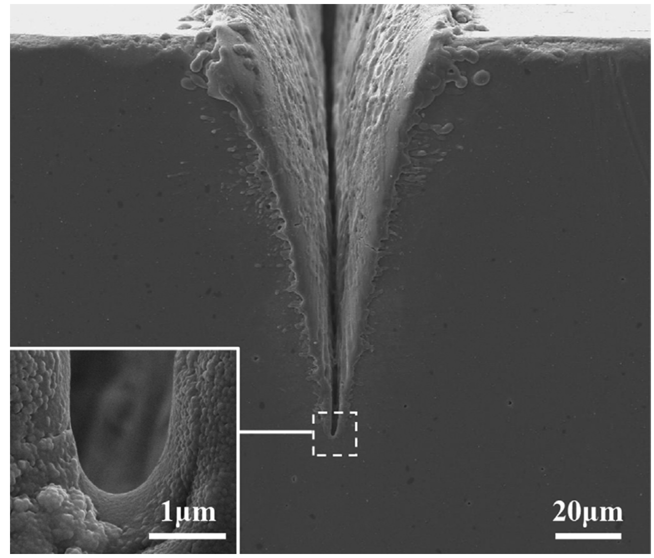

Fig 1. SEM image of ceramic bar with an ultra-sharp V-notch. The enlarged view shows that the V-notch tip radius is less than 1 μm and no microcrack is found near the V-notch root.

2.4. BI method

A Vickers indentation was made at the center of the 2 mm × 22 mm surface first, taking care to align the pyramidal edges with respect to the longitudinal axis for the bar specimens, and then a sharp single edge pop-in precrack was developed by applying the BI technique [20,22]. A dye-penetration technique was adopted [35] and the crack depths were measured optically on both side surfaces.

2.5. Fracture toughness tests

Fracture toughness was determined by the three-point SEVNB method (Model 5569, Instron, China) with a span of 16 mm and a cross head speed of 0.05 mm·min^{-1}. At least six test bars were used to get the average values. The K_{Ic} value was calculated in accordance with ASTM E 1820-13 standard [36]:

$$K_{Ic} = Y\left[\frac{P_{max}S_0}{(BB_N)^{1/2}W^{3/2}}\right] \quad (1)$$

where P_{max} is the fracture load; S_0 is the span width (16 mm); a is the notch depth; B is the sample width (2 mm); W is the sample height (4 mm); B_N is the minimum thickness measured at the roots of the side grooves; Y is a dimensionless correction factor based on the ratio of a/W, which is decided by:

$$Y = \frac{3\left(\frac{a}{W}\right)^{1/2}\left[1.99-\left(\frac{a}{W}\right)\left(1-\frac{a}{W}\right)\left(2.15-3.93\left(\frac{a}{W}\right)+2.7\left(\frac{a}{W}\right)^2\right)\right]}{2\left(1+2\frac{a}{W}\right)\left(1-\frac{a}{W}\right)^{3/2}} \quad (2)$$

3. Results and discussion

Fig. 2 shows the microscopic morphologies of U-groove and V-notches fabricated by traditional and laser notching methods, respectively. The notch tip radius of U-groove prepared by wire cutting machine and/or diamond wheels is ~100 μm. Sharper notches can be obtained by razor blade method. However, the fabrication of notches with tip radius less than 10 μm is a great challenge. In the case of laser notching, the notch tip radius is less than 1 μm and