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Short communication

Vickers indentation crack analysis of solid-phase-sintered silicon carbide ceramics

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Abstract

Solid-phased-sintered silicon carbide ceramics were prepared by traditional process with carbon and boron carbide as additives. The crack initiation and the profiles of the samples after Vickers indentation were observed in order to analyze the crack system. The typical characterizations of Vickers indentation, crack length c and diagonal $2a$ were used to determine the crack system. The surface cracks appear when the indentation load surpasses 0.1–0.2 N or the indentation diagonal length is over 3 μm. Clear and typical half-penny cracks formed after 10 N or above indentations. Within the 3–10 N range, the expected radial crack was not observed; but the relation between the crack length and indentation load told that the crack system after 3–10 N indentation is still half-penny. As to the critical c/a value for crack system transition, if it exists, it does not exceed 2.2. The fact that the toughness by IF method after 3–10 N strongly differs from that after 10–100 N may suggest that the determination of fracture toughness by indentation fracture method is not related to crack system. In addition, it is found that a more precise fracture toughness test of SSiC ceramics by IF method requires an indentation force larger than 10 N.

Keywords: Indentation crack; Crack initiation; Indentation fracture method; Fracture toughness

1. Introduction

Ceramic materials are generally brittle; in ceramics, stress concentrations generated around defects cannot easily be relaxed by plastic flow. Fracture toughness, K_{IC}, represents a key parameter in the definition of damage resistance and material strength, especially in the application of brittle ceramic materials. The indentation fracture (IF) method proposed by Lawn et al. [1–4] is a quick and effective method for fracture toughness determination, which well suits brittle materials of low fracture toughness [5–7]. In practice, the indentation crack only emerges at proper indentation loads [8], and the crack system should be identified firstly [9–11], based on which the proper equation can be determined; any wrong equation choice may result in a dramatic deviation.

The proper indentation load for IF toughness test is usually inferred from the crack system, which transits from radial to half-penny with the increasing indentation load. For this crack system transition, the c/a value, ratio of the crack length $2c$ and the indentation diagonal length $2a$, is an important parameter. Niihara et al. [12] proposed that there is a c/a threshold in the crack transition. It was estimated that the critical c/a ratio was about 2.5 regardless of material system; however, other investigations [5,6,13] proved that it is not a constant. For example, the critical c/a value is about 2 for alumina, and 3 for 3 mol% yttria tetragonal zirconia polycrystal (3Y-TZP) [13]. In addition, it varies even in the same material. Miyazaki et al. [14] demonstrated that the critical c/a value of silicon nitride ceramics fluctuates around 2 with different microstructures.

Solid-phase-pressureless-sintered silicon carbide ceramic (SSiC for short) has been widely used as structural material for its excellent natures. But its toughness, just as other

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ceramics, is the major shortcoming. The crack system of silicon carbide ceramics were not systematically investigated as that of silicon nitride ones [5,6]. In this work, the Vickers indentation of SSiC ceramics was studied by microscopy with the help of decoration method. Crack initiation and crack profile geometry were observed by SEM, and the typical characterization of Vickers indentation, crack length and half diagonal length, were also investigated to analyze the crack initiation and crack system for SSiC ceramics.

2. Experimental procedure

2.1. Sample preparation

Commercially available silicon carbide (SiC) powders were firstly mixed with carbon and boron carbide as sintering additives by general ball-milling. Ethanol was used as the mixing media. The mixtures were then dried, sieved, dry-pressed and cold-isostatically pressed under a pressure of 200 MPa. Finally, as-prepared green bodies were sintered at 2200 °C for 1 h in an argon flow of atmosphere pressure.

2.2. Test procedure

All the prepared SSiC samples were firstly cut, grinded, and mirror-polished before measurements. The fracture toughness (K_{IC}) and Vickers hardness (H_V) of SSiC ceramics were measured by indentation fracture method with a hardness tester (Wilson Tukon 2100B). The fractured and polish-etched surfaces of SSiC ceramics were observed by scanning electronic microscope (SEM, JSM-6700 F) to determine the microstructures. And to investigate the crack initiation and crack profile geometries, the Vickers indenter was firstly loaded on mirror-polished surfaces of SSiC samples with different indentation loads varying in the range of 0.1–100 N, and then the indentation topographies and crack profile geometries at various indentation loads were observed by SEM (Hitachi TM-1000). To observe the crack profile geometries distinctly, the decorating technique was adopted to the indented regions immediately after unloading as follows [13–15]: drop saturated lead acetate solution on the indentation, leave the droplet on the surface for 3–4 h until the solution completely crystallizes, clear the residual lead acetate after a 5–10 min drying at over 100 °C, break the sample bar at the indentation and again observe the crack profiles by SEM. At least 10 test bars were employed in each test to minimize the dispersion of brittle ceramics.

3. Results and discussion

3.1. Crack initiation of SSiC ceramics

Fig. 1 shows the SEM images of the fractured surface and polish-etched surface of SSiC sample. The microstructure of the as-prepared SSiC ceramics homogeneously consists of equiaxed grains of an average size of 3–5 μm and is free of pore. The Vickers hardness of SSiC is 24.2 GPa, and the fracture toughness calculated by Miyoshi's equation [16] is 2.8 MPa m$^{1/2}$ after 20 N indentation by Vickers indenter.

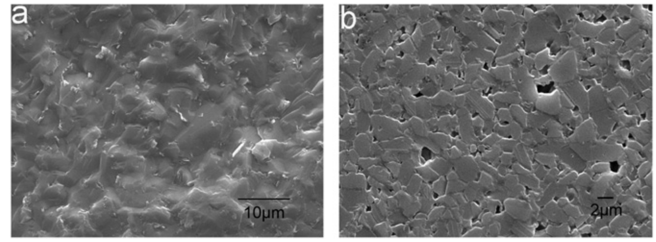

Fig. 1. SEM images of SSiC ceramics: (a) fractured surface; (b) polish-etched surface.

Fig. 2 presents the SEM images of Vickers indentation topography at various loads. As shown in Fig. 2(a), it is difficult to discern between the 0.1 N indentation and a residual carbon dispersed in the matrix because their dimensions are comparative and no crack emerges from the indentation. Both Fig. 2(b) and (c) demonstrates the indentations by 0.2 N indentation load; although indentations of the two are identical in indentation diagonal length (about 3 μm), the extended cracks are quite different. The cracks are absent, as shown in Fig. 2(b), or present, as shown in Fig. 2(c), from time to time. Fig. 2(d) and Fig. 2(e) represents the indentations by 0.5 N and 1 N respectively, which show that the indentation diagonal lengths are about 5 μm and 8 μm, and the extended cracks are obvious. It is therefore reasonable to conclude that the surface crack of SSiC ceramics initiates when the Vickers indentation load lies between 0.1 N and 0.2 N or the diagonal length of indentation reaches to 3 μm. These are the very critical conditions for the Vickers crack initiation on SSiC surfaces.

Cracks are inevitable beyond the critical condition; otherwise, there only emerge indentations without any extended cracks on the material surface. Lawn et. al. [2,8] proposed the critical conditions for the cracks initiation as follows:

$$a_c = \Theta (K_{IC}/H_v)^2 \tag{1}$$

$$P_c = \Theta\, \alpha_\theta H (K_{IC}/H_v)^4 \tag{2}$$

where a_c and P_c are the critical indentation half-diagonal length and the critical indentation load for the cracks initiation respectively, and both Θ and α_θ are dimensionless factors correlative to crack geometry, and K_{IC} represents fracture toughness.

Based on abovementioned equations, Lawn et al. constructed a universal deformation-fracture diagram by normalization method for various materials including silicon carbide, silicon nitride, and boron carbide, and so on [1,2]. According to the diagram, it is extrapolated that $\Theta = 110$ and $\alpha_\theta = 0.16$ for SSiC ceramics. Substituting $H_V = 24.2$ GPa and $K_{IC} = 2.8$ MPa m$^{1/2}$ into Eqs. (1) and

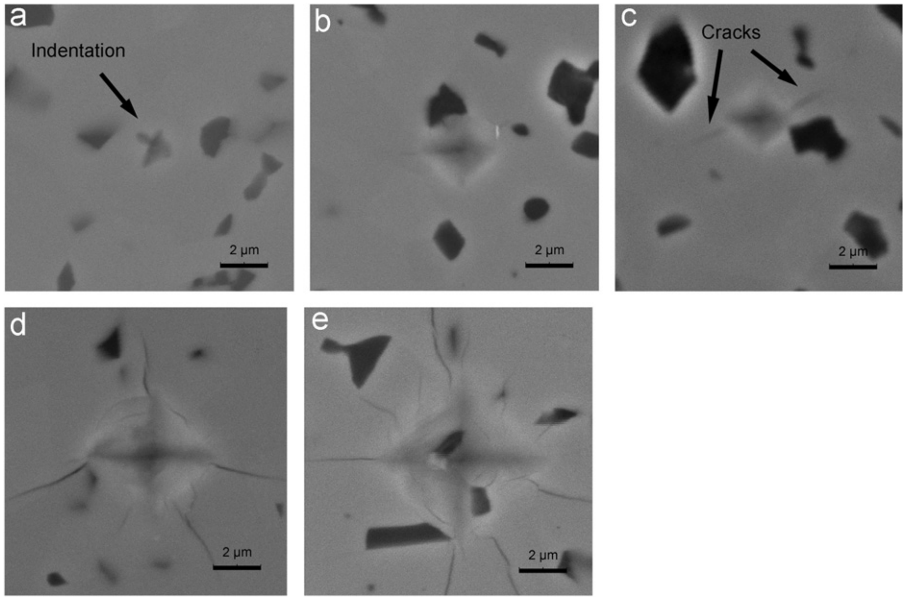

Fig. 2. SEM images of Vickers topography of SSiC ceramics at different load: (a) 0.1 N; (b), 0.2 N; (c) 0.2 N; (d) 0.5 N; (e) 1 N.

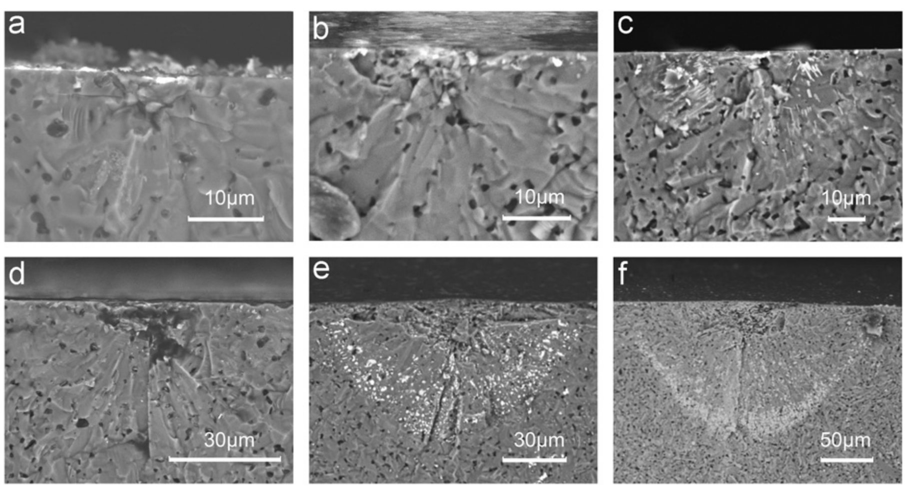

Fig. 3. SEM images of SSiC ceramics crack profile geometries at different loads: (a) 3 N; (b) 5 N; (c) 7 N; (d) 10 N; (e) 20 N; (f) 50 N.

(2), a_c and P_c of SSiC ceramics are obtained as 1.5 μm and 0.08 N, respectively. It means that some cracks can be expected extending from the indentation when the indentation half-diagonal length a is larger than 1.5 μm or the indentation load P surpasses 0.08 N. The measured critical indentation diagonal length $2a_c$ is about 3 μm, as the SEM results shown in Fig. 2 which is in good accordance with the calculated value; and the critical indentation load P_c of 0.1–0.2 N, is also in good agreement with the calculated value in consideration of the discreteness of ceramics materials.

3.2. Crack profile analysis of SSiC ceramics

The fracture toughness of ceramic materials can be reliably calculated from semi-empirical equations by the indentation fracture method only if the indentation cracks can be recognized. Therefore, it is important to determine the crack system before further measurements.

Vickers crack profiles of SSiC ceramics at various indentation loads were decorated by saturated lead acetate solution, and the results are shown in Fig. 3. The crack profiles in Fig. 3(d)–(f) show that the crack are clearly half-penny system, and that in (a)–(c) are not clearly enough as in (d)–(f) to tell their types, but the boundaries seem to be half-penny. Those results show that Vickers crack of SSiC ceramics is typical half-penny after 10 N and above indentations. The cracks after indentations smaller than 10 N are difficult to discern. The typical radial crack is not observed at the load range from 3 N to 100 N, therefore the transition of crack systems from radial to half-penny in SSiC is not observed.

The crack systems can also be distinguished by the relationship between the crack length c and indentation

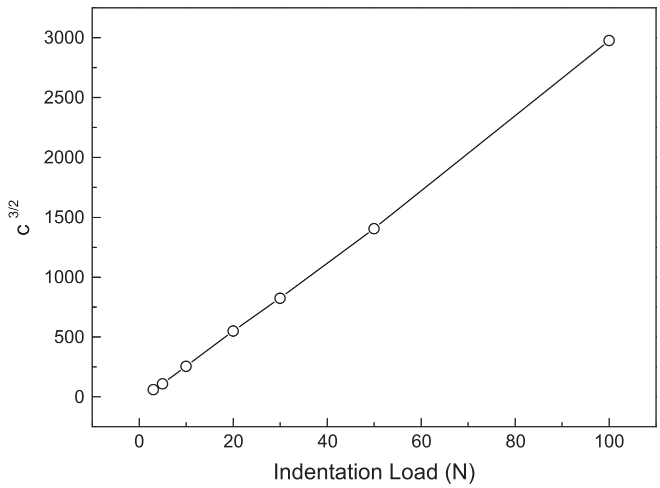

Fig. 4. Relationship between crack length c and indentation load P of SSiC ceramics.

load P [17,18]. The research on Al–Ni abrasive materials and soda-lime glasses [18] showed that the c and P satisfy the following equation for half-penny crack system: $c=AP^{2/3}$, which means $c^{3/2}\propto P$. Fig. 4 demonstrates the relationship of extended crack length c and indentation load P of SSiC ceramics. The good linear dependence between crack length $c^{3/2}$ and indentation load P is consistent with Lube's results [6] that good credibility of the crack length measurement can be expected; more importantly, the corresponding of $c^{3/2}\propto P$ indicates that the cracks extended from the 3–100 N indentations of both SSiC and LSiC ceramics are all half-penny crack systems according to the conclusion from [18]. It is correspondence with the results in Fig. 3 that the cracks after 10 N and above loads are typical half-penny. And the cracks after 3–10 N loads should be half-penny system too.

Pajares et al.[13] explained the crack systems by the three-zone model that the crack profiles depend on both the hydrostatic stress field originating from the plastic deformation underneath the Vickers indentation and the residual stress field resulting from the indentation deformation. The crack is kidney-shaped radial crack when the residual stress field is dominant and the crack becomes half-penny when the hydrostatic stress field is predominant. As the indentation increases, the kidney-shaped radial cracks extend along the surface and in depth, and the coplanar kidney cracks join together and turn to half-penny crack at high enough indentation loads. They suggested a transition from radial to half-penny geometry when the load P or the ratio c/a surpasses a minimum value as follows:

$$P^{*}=\left(\frac{8m^{6}}{\chi^{4}}\right)\cdot\left(\frac{K_{IC}^{4}}{H_{V}^{3}}\right) \qquad (3)$$

where P^{*} is the critical indentation load of crack transformation, m ($m=c/a$) the hydrostatic stress parameter determined by certain c/a range, χ a constant representing the residual stress field, and K_{IC} and H_V the fracture toughness and hardness, respectively. The equation explains the relation between the crack system transition and the ratio c/a.

The indentation half-diagonal length a, crack length c, and characteristic value c/a of SSiC ceramics at different indentation loads P are shown in Fig. 5, by which it is found that the characteristic c/a value is 2.2 at 3 N, 2.5 at 5 N, 2.7 at 7 N and 2.9 at 10 N. The fact that the radial crack system is not observed suggests that the crack system should not be determined by the c/a value for SSiC ceramic. And the critical c/a cannot be told, but it should be no more than 2.2 for SSiC ceramics if it exists.

The fracture toughness K_{IC} evaluated from Miyoshi's equation [16] by IF method of SSiC ceramics as a function of the indentation load was shown in Fig. 6. The fracture toughness values after 10 N and above indentation loads are close to a constant value of 2.8 MPa m$^{1/2}$, which well agrees with the fracture toughness of sintered alpha SiC [19] 2.6–2.8 MPa m$^{1/2}$. But the toughness at smaller indentation

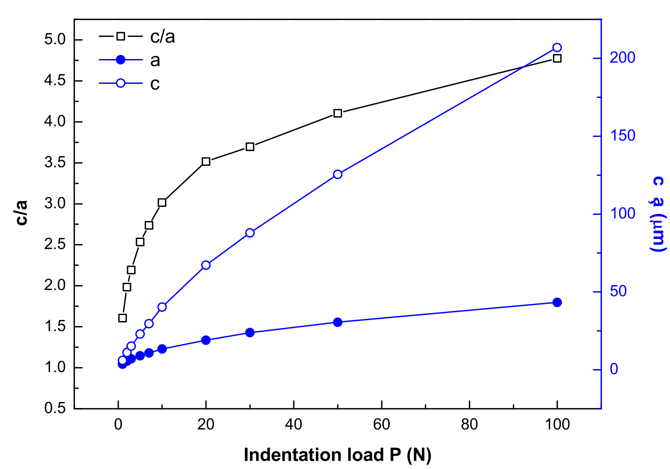

Fig. 5. Typical indentation size of SSiC ceramics at different indentation loads.

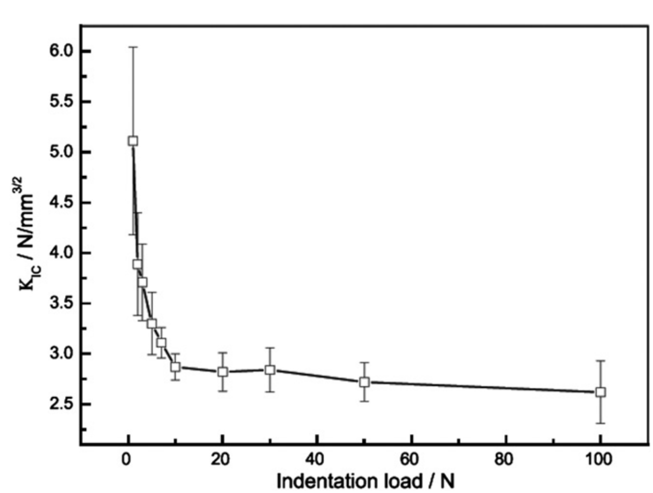

Fig. 6. Fracture toughness of SSiC ceramics by IF method as a function of indentation load.

loads decreases steeply as the load increases from 1 N to 10 N and deviates dramatically from the steady value. Although the crack system after 3–10 N indentations are the same half-penny as after 10–100 N indentations, as shown in Fig. 4, the toughness calculated from the same Miyoshi's equation differ strongly as shown in Fig. 6. Therefore, it is reasonable to conclude that the determination of fracture toughness by IF method is not related to crack system. And as an empirical practice, the loaded force should be more than 10 N for the fracture toughness determination of SSiC ceramics by IF method.

4. Conclusions

SSiC ceramics samples were prepared by traditional process, the microstructure of which is homogeneously consisted of 3–5 μm equiaxed grains. The surface cracks appear when the indentation load surpasses 0.1–0.2 N or the indentation diagonal length is over 3 μm. Clear and typical half-penny cracks formed after 10 N or above indentations. Within the 3–10 N range, the expected radial crack was not observed; but the relation between the crack length and indentation load told that the crack system after 3–10 N indentation is still half-penny. As to the critical c/a value for crack system transition, it does not exceed 2.2 if it exists. The fact that the toughness by IF method after 3–10 N strongly differs from that after 10–100 N may suggest that the determination of fracture toughness by indentation fracture method is not related to crack system. In addition, it is found that a more precise fracture toughness test of SSiC ceramics by IF method requires an indentation force larger than 10 N.

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References

[1] B.R. Lawn, A.G. Evans, D.B. Marshall, Elastic/plastic indentation damage in ceramics: the median/radial crack system, Journal of the American Ceramic Society 63 (9–10) (1980) 574–581.

[2] B. Lawn, in: Fracture of Brittle Solids, Senior Education Press, Beijing, 2010.

[3] B.R. Lawn, D.B. Marshall, Hardness, toughness, and brittleness: an indentation analysis, Journal of the American Ceramic Society 62 (7-8) (1979) 347–350.

[4] A.G. Evans, E.A. Charles, Fracture toughness determinations by indentation, Journal of the American Ceramic Society 59 (7-8) (1976) 371–372.

[5] H. Miyazaki, H. Hyuga, Y.-i. Yoshizawa, K. Hirao, T. Ohji, Crack profiles under a Vickers indent in silicon nitride ceramics with various microstructures, Ceramics International 36 (1) (2010) 173–179.

[6] L. Tanja, Indentation crack profiles in silicon nitride, Journal of the European Ceramic Society 21 (2) (2001) 211–218.

[7] H. Miyazaki, H. Hyuga, Y.-i. Yoshizawa, K. Hirao, T. Ohji, Relationship between fracture toughness determined by surface crack in flexure and fracture resistance measured by indentation fracture for silicon nitride ceramics with various microstructures, Ceramics International 35 (1) (2009) 493–501.

[8] B. Lawn, A. Evans, A model for crack initiation in elastic/plastic indentation fields, Journal of Material Science 12 (1977) 2195–2199.

[9] B. Lawn, R. Wilshaw, Indentation fracture: principles and applications, Journal of Material Science 10 (1975) 1049–1081.

[10] G.Y. Lin, R. Lakshminarayanan, D. Shetty, Contact damage initiation in silicon nitride in Hertzian indentation: role of microstructure, Journal of Material Science 42 (2007) 3508–3519.

[11] Z. Li, A. Ghosh, A.S. Kobayashi, R.C. Bradt, Indentation fracture toughness of sintered silicon carbide in the palmqvist crack regime, Journal of the American Ceramic Society 72 (6) (1989) 904–911.

[12] K. Niihara, R. Morena, D.P.H. Hasselman, Evaluation of K_{Ic} of brittle solids by the indentation method with low crack-to-indent ratios, Journal of Materials Science Letters 1 (1) (1982) 13–16.

[13] A. Pajares, F. Guibertea, F. Cumbrera, R. Steinbrech, A. Dominguez-Rodriguez, Analysis of kidney-shaped indentation cracks in 4Y-PSZ, Acta Materialia 44 (1996) 4387–4394.

[14] S.L. Jones, C.J. Norman, R. Shahani, Crack-profile shapes formed under a vickers indent pyrami, Journal of Materials Science Letters 6 (6) (1987) 721–723.

[15] V.M. Sglavo, P. Pancheri, Crack decorating technique for fracture-toughness measurement in alumina, Journal of the European Ceramic Society 17 (14) (1997) 1697–1706.

[16] T. Miyoshi, N. Sagawa, T. Sassa, Study on fracture toughness evaluation for structural ceramics, Transactions of the Japan Society of Mechanical Engineers Series A 51 (471) (1985) 8.

[17] D.K. Shetty, I.G. Wright, P.N. Mincer, A.H. Clauer, Indentation fracture of WC-Co cermet, Journal of Materials Science 20 (5) (1985) 1873–1882.

[18] E. Breval, G.C. Dodds, N.H. Macmillan, The hardness, stiffness and toughness of diphasic abrasive materials prepared by sol–gel techniques, Materials Research Bulletin 20 (4) (1985) 413–429.

[19] G.D. Quinn, J.A. Salem, Effect of lateral cracks on fracture toughness determined by the surface-crack-in-flexure method, Journal of the American Ceramic Society 85 (4) (2002) 873–880.