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Ceramics International 33 (2007) 315–320

CERAMICS INTERNATIONAL

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Evaluation of fracture resistance of ceramics: Edge fracture tests

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Received 17 June 2005; received in revised form 27 July 2005; accepted 20 September 2005
Available online 28 February 2006

Abstract

Advanced oxide and nonoxide ceramics were tested by edge flaking with different indenters. Flaking toughness values obtained with the Rockwell indenter were shown to be practically proportional to fracture toughness values measured by the single edge V-notched beam (SEVNB) method. The discussed edge fracture (EF) method enabling the determination of fracture resistance of ceramics in small-size specimen tests was demonstrated to be appropriate for materials technology as well as for the evaluation of ceramics for dentistry, cutting tools, and like uses. Emphasis was placed on the results of investigation of chip scars formed upon flaking off the specimens with the Rockwell, Vickers, and Knoop indenters.

Keywords: C. Fracture; D. Conventional ceramics; Edge fracture; Flaking toughness; Indentation

1. Introduction

Fracture of brittle materials upon concentrated loads attracted attention already in the Paleolithic Period, when stone tools were first developed. This approach was also used for the choice of the best stones for tool production, which became the starting point for fracture resistance tests.

A study [1] was a new step in this direction; its authors revealed the linear relationship between chip sizes (chip width-to-height and thickness-to-height ratios are approximately constant) at the same loads applied to the edge of the material. They proposed [2] to indent a rectangular specimen with the polycrystalline Rockwell indenter using increasing loads at different distances from the edge until a chip breaks off, determined the linear relation between the load P_f required to produce a chip and the distance d of the indenter from the edge, and suggested the ratio P_f/d as a measure of the edge toughness M of the material. One of the authors [3] proposed to use the slope of the fracture load P_f versus edge distance d diagram to determine this value. These tests would require a special test machine because the loading point on indentation must be chosen very precisely. For different brittle materials under study (from Crown glass to tungsten carbide) the edge toughness M versus critical plane strain energy release rate G_{Ic} relationship was also established. Authors [4] used the Rockwell and Knoop indenters to investigate not only ceramics and hard metals but also brittle tool steels. They confirmed the validity of the M–G_{Ic} relationship and noticed that the K_{Ic}–G_{Ic} relationship was not observed. Several dental ceramics were extensively studied elsewhere [5]. The tests were performed under small loads using a diamond cone. The initial portions of P_f–d diagrams were nonlinear. Sintered Si_3N_4 ceramics for valves of internal combustion engines were also investigated [6,7]. Studies of the effect of specimen edge shapes on edge toughness resulted in the modification of their design.

Known data, while very interesting and useful, demonstrate that the M–G_{Ic} relationship has no physical substantiation. It is not observed for ceramics [8,9] but holds for different brittle materials (e.g., test results [2,3]). A known procedure does not take account of differences in the mechanical behavior of ceramics [10] and other brittle materials, fracture patterns in the zone of the indenter contact with the specimen as well as the real fracture surface area formed upon flaking. Therefore, a more advantageous edge fracture method was developed [9]. Its efficiency and suitability for testing different ceramic materials was verified in the present investigation.

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0272-8842/$32.00
doi:10.1016/j.ceramint.2005.09.017

2. Materials and methods

2.1. Ceramics

Gas-pressure-sintered silicon nitrides GPSSN, sintered silicon carbides EKasic® C as well as sintered zirconia ceramics of different structure and composition were the major materials investigated in this study (Table 1). To compare the above method with the single edge V-notched beam (SEVNB) one, linear elastic ceramics were tested (similar ceramics are usually used in Round Robin fracture toughness tests [13].

All these ceramics were fine- and coarse-grained materials of rather high homogeneity. The exception was commercial EKasic® C ceramics (ESC Ceramics GmbH & Co. KG, Germany) with grain sizes up to 1500 μm and inelastic TS-grade ceramics (Nilcra Incorp., Australia).

2.2. Test method

According to our procedure, a loading point on the specimen is first chosen by the naked eye (or by means of a magnifying glass), edge flaking of ceramics is effected by indentation, and the peak load P_f at the moment of chip formation is determined. Then the fracture distance L (Fig. 1), which is equal to the chip thickness, is accurately measured on the specimen using the micrometer eye-piece of an Olympus BX 51M optical binocular microscope (1000×). The slope of the regression line for data points (P_f–L diagram) obtained under constant experimental conditions was used as the characteristic of the flaking toughness E_t.

The Rockwell, Knoop, and Vickers diamond indenters were used to apply the load at the edge of the polished rectangular specimen with an edge radius of about 10–20 μm and cross-sections from 3 mm × 3 mm to 9 mm × 10 mm. The specimens were tested at varying fracture distances L from the edge (Fig. 2) and P_f values, which changed accordingly, were recorded using a computer. The indenters were placed in the moving rod of a CeramTest device of our design [14], mounted on a multipurpose compression/tension test machine (cross-head speed in experiments was constant and equal to 0.5 mm/min). The specimens were fixed in place with the holder of an X–Y table installed on the rigid load cell of this device. Lateral cracks and a heave of the specimen surface near their edges did not appear on indentation as well as chips flaked off from the specimens did not hack to pieces.

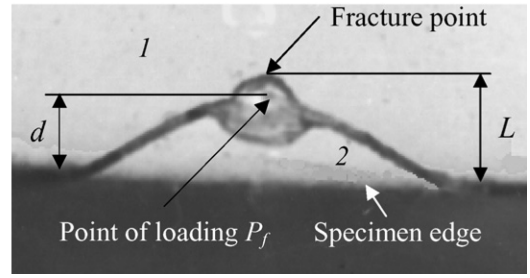

Fig. 1. Edge flaking of a hot-pressed silicon nitride [9] specimen (Rockwell indenter, fracture load, P_f = 83 N, fracture distances, L = 0.18 mm): 1—specimen, 2—chip.

SEVNB fracture toughness values were determined according to a known procedure [14].

3. Results and discussion

The first step of our investigation based on the use of the Rockwell indenter was confirmed the existence of the linear P_f–L relationship for different ceramic materials. Sintered Al_2O_3/(Y, Nb, Fe)-TZP (K_{Ic} = 5.9 MPa m$^{1/2}$), self-bonded SiC + 20% Si (K_{Ic} = 2.4 MPa m$^{1/2}$) as well as GPSSN ceramics were used in these experiments. In accordance with the obtained results (Fig. 3), the P_f–L relationship is independent of P_f values and therefore flaking toughness E_t can be related to the mechanical properties of the material. It should be noted that the edge toughness M was proposed using a similar approach [2].

The second step of our investigation consisted in the comparison of experimental and published results of flaking toughness determinations. Ceramics similar to those investigated by other authors were tested using the Rockwell indenter and closely related data for several materials were obtained [9]. The studies on Prozyr zirconia ceramics, for instance demonstrate that our L-based measurement results can be sufficiently consistent with data derived by measuring the value d on a commercially available device designed for this purpose (Fig. 4).

The third step involved the evaluation of the effect of indenter types on test results. To do this, the majority of

Table 1
Characteristics of ceramic materials

Ceramics	Nomenclature and reference	Brittleness measure χ [9]	Elastic modulus (GPa)	Strength (MPa)	Fracture toughness (SEVNB) (MPa m$^{1/2}$)
Scandia	Sc_2O_3 [11]	1	218	110	1.49
Alumina	Al_2O_3-2 [12]	1	232	322	2.93
Zirconia	Prozyr (Y-PSZ) [5,9]	1	210	1300	4.90
	Mg-PSZ (TS-grade) [9,10]	0.42	206	521	10.40
	Y-TZP [9]	1	206	521	7.40
Silicon nitride	GPSSN [9,13,14]	1	320	920	5.40
	Si_3N_4 [12]	1	273	486	4.32
Silicon carbide	EKasic® C [9]	1	430	389	2.80

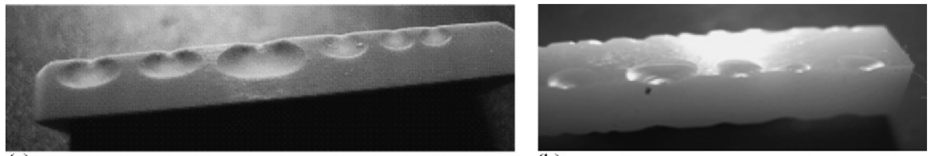

Fig. 2. Chip scars on GPSSN (a) and Prozyr specimens (3 mm × 4 mm cross-section).

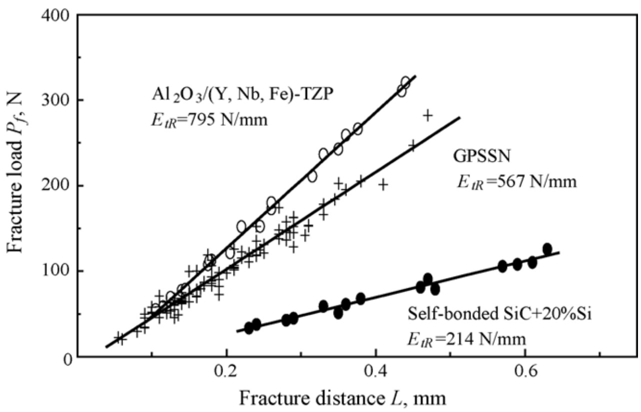

Fig. 3. Fracture load P_f vs. fracture distance L diagrams for different ceramics.

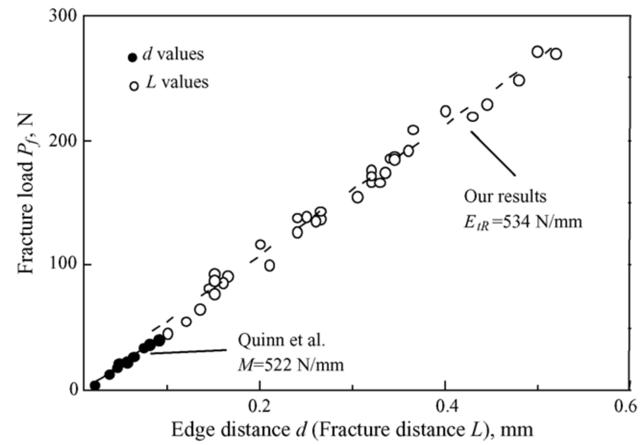

Fig. 4. Comparison of fracture load P_f vs. edge distance d (fracture distance L) diagrams for Prozyr ceramics tested in [5] and in our laboratory.

materials under study were tested with different indenters. These results demonstrate that flaking toughness values decreased with stress concentrations in the fracture zone (Fig. 5a). For silicon carbide ceramics, characterized by the wide scatter of the data, it appeared to be narrower. Such a result does not correspond to [4]. Therefore, an additional investigation of EKasic® C ceramics was undertaken. To obtain data (Fig. 5b), these ceramics were studied on the specimens with a cross-section of about 3 mm × 3 mm, therefore, one can infer that the wide scatter of experimental data is associated with size effects (chip scars were higher than half the specimen thickness, as is shown in Fig. 2), hence, thicker specimens (9 mm × 10 mm) were tested. As is seen in Fig. 6, the picture does not change: the same E_{tR} value and the same scatter of the results. But it should be noted that the scatter of the data for other similar ceramics [4] was wider than that observed in our experiments, which can be associated with the specific features of the test method used (see Section 1). It is significant that any changes in the scatter of the results for GPSSN (Fig. 5a) and Y-PSZ ceramics were not observed, although flaking toughness values were different. On this basis, it is suggested to use the following terms: Rockwell flaking toughness E_{tR}, Knoop flaking toughness E_{tK}, and Vickers flaking toughness E_{tv}.

The fourth step referred to the examination of the relationship between chip scar sizes on the specimens. The results of this investigation for elastic homogeneous ceramics demonstrated a linear proportionality between maximum width-to-fracture length and height-to-fracture length ratios for them (Fig. 7). It means that the EF method is effective at any chip sizes, when flaking toughness values are measured. Thus, it is appropriate to substitute W measurements for L ones because such measurements are easier to perform more precisely by an optical microscope.

In addition to ceramics listed in Table 1, a hot-pressed silicon nitride particulate ceramic composite with 10% TiN (elastic modulus 320 GPa) [14] was also studied. The fracture toughness of this material was different (4.8 and 5.15 MPa m$^{1/2}$) in the direction A (parallel to the hot pressing axis) and the direction B

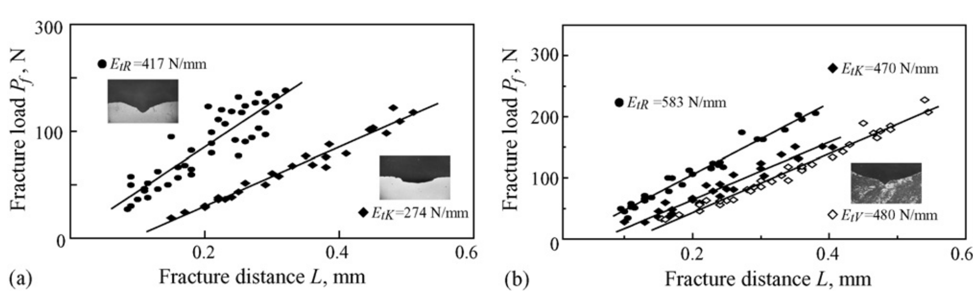

Fig. 5. Fracture load P_f vs. fracture distance L diagrams for EKasic® C (a) and GPSSN (b) specimens obtained with different indenters.

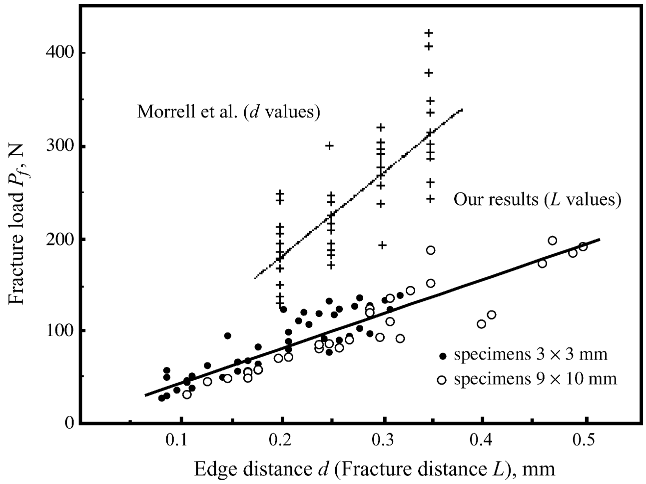

Fig. 6. Fracture load P_f vs. fracture distance L diagrams for EKasic® C ceramics (specimens of different sizes) and data [4].

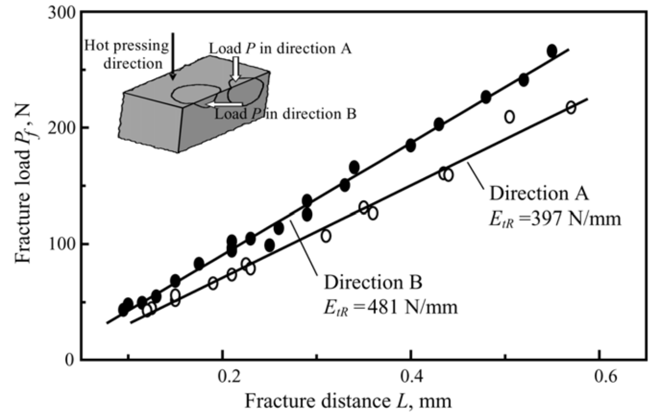

Fig. 8. Fracture load P_f vs. fracture distance L diagrams for a silicon nitride/titanium nitride particulate ceramic composite (different loading directions).

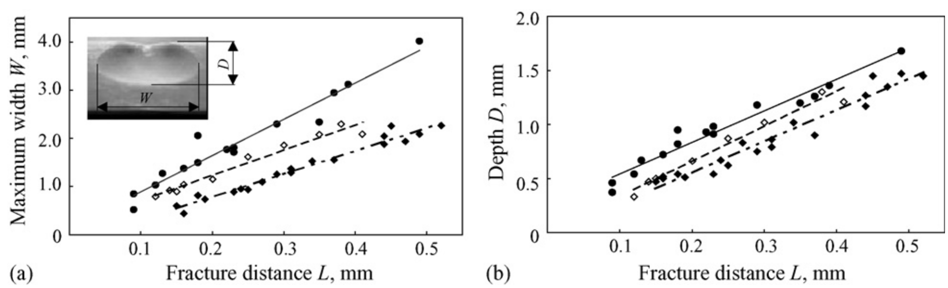

Fig. 7. Maximum chip scar width W (a) and chip scar depth D (b) vs. fracture distance L diagrams of GPSSN specimens tested with different indenters (●: Rockwell, ◇: Vickers, and ◆: Knoop).

(normal to the hot pressing axis), accordingly. Such an effect is also typical of flaking toughness, as is shown in Fig. 8. It is interesting to note that in case of a silicon nitride/silicon nitride-titanium nitride laminar ceramic composite with a compressed Si_3N_4 outer layer [14], the relationship between flaking toughness in the A and B directions is opposite. Tensile stresses in the compressed outer layer contribute to its fracture under loading in the direction B.

Considerable study has been given to chip scars on the tested specimens because they control the accuracy of the data obtained in this investigation. As is seen, symmetrical chip scars (Fig. 2) can be obtained if the edge of the specimen is rectangular, its surface is polished, its loading plane is perpendicular to the indentation direction. However, in a lot of cases chip scars look differently. For instance, chip scars formed on hot-pressed Si_3N_4 (Fig. 1) were similar to those shown in [4] for PM tool steel (64 HRC), the shape of grains governed fracture patterns of EKasic® C ceramics (Fig. 9), etc. Based on the results of this investigation and published data, it is impossible to examine all the factors controlling edge fracture patterns in different ceramic materials.

Mention should also be made of some unintelligible effects observed during the experiments, e.g., nonlinearity of fracture

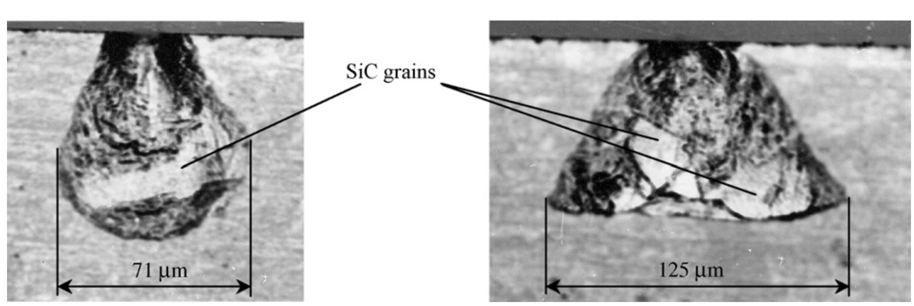

Fig. 9. Chip scars on an EKasic® C specimen.

Table 2
Comparison of test methods

Method	Choice of a loading point	Tool for fracture	Characterization of the tested specimen	Analysis	Results
Paleolithic	Naked eye	Stone in hand	Visual examination of chip scars	Choice of more fracture-resistant stones	Production of high-quality stone tools
Known [2]	Microscope of a special test unit	Indenter in a special test unit	–	Calculation of edge toughness	Data of edge fracture resistance evaluation
Edge fracture	Naked eye (or a magnifying glass)	Indenter in a standard test unit	Measurement of chip scar sizes	Calculation of flaking toughness	Data for fracture toughness evaluation

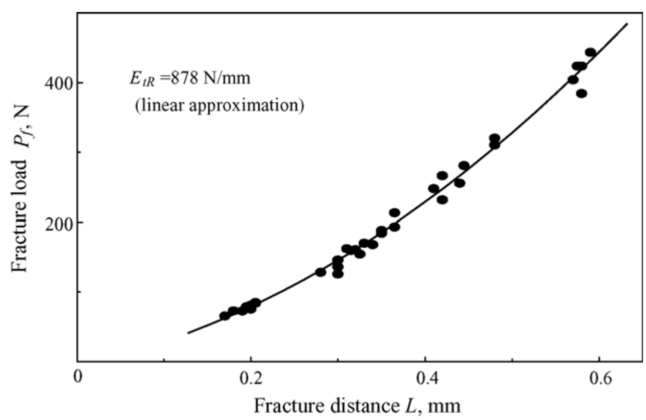

Fig. 10. Fracture load P_f vs. fracture distance L diagram for Mg-PSZ ceramics.

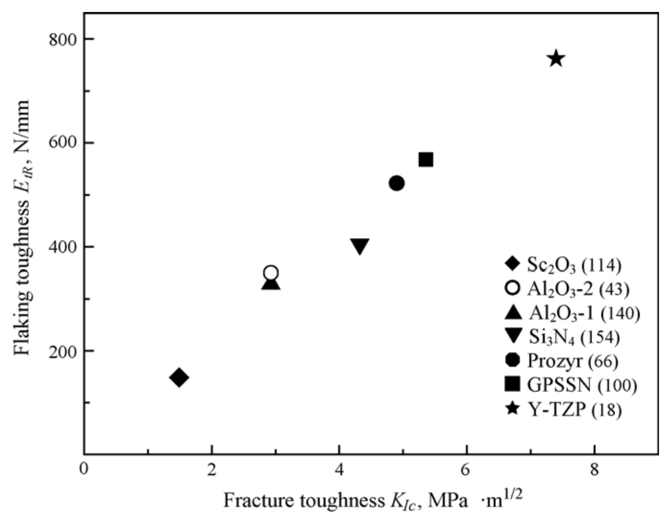

Fig. 11. Flaking toughness E_{tR} vs. fracture toughness K_{Ic} diagram for the ceramics under study (characters in brackets are the number of tests).

load P_f versus fracture distance L diagrams (Fig. 10). As a result of their linear approximation, E_{tR} = 878 N/mm can be obtained. If the P_f–L diagram is divided into 0.15–0.30 mm, 0.30–0.45 mm, and 0.45–0.60 mm pieces, E_{tR} = 550, 891, and 1040 N/mm are derived. If the P_f–L diagram is divided into a larger number *of* pieces or differentiated, the obtained diagram would be similar to the R-curve for these ceramics [10]. This problem will be investigated in the immediate future but it is already clear that the EF method can provide new reliable data on the fracture resistance of ceramics.

Before proceeding to the discussion of the results, it would be appropriate to compare the EF method with those described elsewhere [2] and probably used in the Paleolithic Period (Table 2).

Summarizing the investigation results, it might be well to evaluate the flaking toughness E_{tR} versus fracture toughness K_{Ic} relationships for linear elastic and single phase ceramics under study (Fig. 11). In this case the validity of the SEVNB method is obvious since it makes use of fracture mechanics concepts [15] based on the model solid body which is homogeneous and linear elastic. These diagrams demonstrate that the flaking toughness E_{tR} versus fracture toughness K_{Ic} relationship is almost linear.

4. Conclusions

The edge fracture (EF) method can easily be put to practical use in any laboratory performing mechanical tests.

It has been shown that the Rockwell flaking toughness E_{tR} versus fracture toughness K_{Ic} relationship is almost linear. Thus, E_{tR} values can be used for the comparative evaluation of the fracture resistance of ceramic materials.

Acknowledgments

The authors are thankful to Professor E. Almond (National Physical Laboratory, UK) for his idea of studying the edge fracture of ceramics, Dr. J. Quinn (American Dental Association Foundation, USA) for her support in launching this investigation, Dr. J. Kubler (EMPA, Switzerland) and Dr. A Rendtel (ESK Ceramics GmbH & Co. KG, Germany) for ceramic specimens as well as Eng. B. Ozersky (Pisarenko Institute for Problems of Strength, Ukraine) for his assistance in the performance of experiments.

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