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Fracture barrier estimation by the edge fracture test method

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Abstract

Known methods for determining the fracture resistance of brittle materials by indentation flaking of the rectangular specimen edge are compared. The edge fracture (EF) test method was chosen as an optimum one. The investigations demonstrated that silicon carbide ceramics, submicrometre alumina, and glasses possessed fracture resistance (F_R) values that can be located above the baseline in the EF base diagram. These results emphasize that the above materials display a higher resistance to the onset of fracture (fracture barrier). This barrier can be estimated by comparing EF test data resulting from Rockwell and Vickers indentations.

Keywords: C. Fracture; D. Glass; Silicon carbide; Alumina; Edge fracture; Fracture barrier

1. Introduction

The fracture behaviour of brittle materials attracts considerable attention of researchers. Different aspects of fracture behaviour are usually examined by indentation flaking (chipping) of the rectangular specimen edge. For such tests, small material quantities would be required, which is important for materials science research, especially for investigation of expensive compositions or optimization of material properties.

Nowadays these investigations (see also [1]) are performed in two different ways (A and B in Fig. 1). The first one employs the fracture parameter, being the distance from the point of applying the indentation load to the specimen edge. This can be performed on a special test unit with the pin-point positioning of an indenter. The second one suggests visual choice of an indentation point on the specimen surface near its edge, its further flaking on a conventional test machine, and microscopic measurement of the distance from the specimen edge to the extreme point on the chip scar (Fig. 2). In all the cases, but [7], the fracture load/adopted fracture parameter ratio is used as the general characteristic of fracture resistance, however, its calculated values do not coincide.

The edge fracture (EF) test method stands out from the others, since it provides the fracture resistance characteristic proportional to fracture toughness values, determined by the single edge V-notch beam (SEVNB) method, when ceramics are consistent with the solid model of linear fracture mechanics [18] (this relation is termed the *baseline* [13]). Fracture resistance values, e.g., for hot-pressed boron carbide [13] and glasses [17], may be higher than those, obeying the above relation, which applies to other elastic dense materials. This fact was earlier overlooked, since the above first fracture stage is usually effected by preparing stress concentrators in test specimens. It is also may stem from the fact that this issue is not treated by linear fracture mechanics [18], based on Griffith's classical work [19], where the propagation of an initial crack is analyzed, while its nucleation has not received mentioning [20].

Since information on this effect may be of practical interest, its investigations have been continued. The results are presented in this communication.

2. Materials and methods

Elastic oxide and nonoxide ceramics and optical quartz glass were chosen for the investigation (Table 1). Their tests were carried out as described in [1]. The fracture toughness (K_{Ic}) was first evaluated by the SEVNB method [23] in three-point flexure of polished rectangular specimens (3-mm × 4-mm cross-section, edge radius less than 30 μm), with a V-notch of a tip radius of about 5 μm. Specimen fragments, formed in these experiments, were used in EF tests. The experiments were

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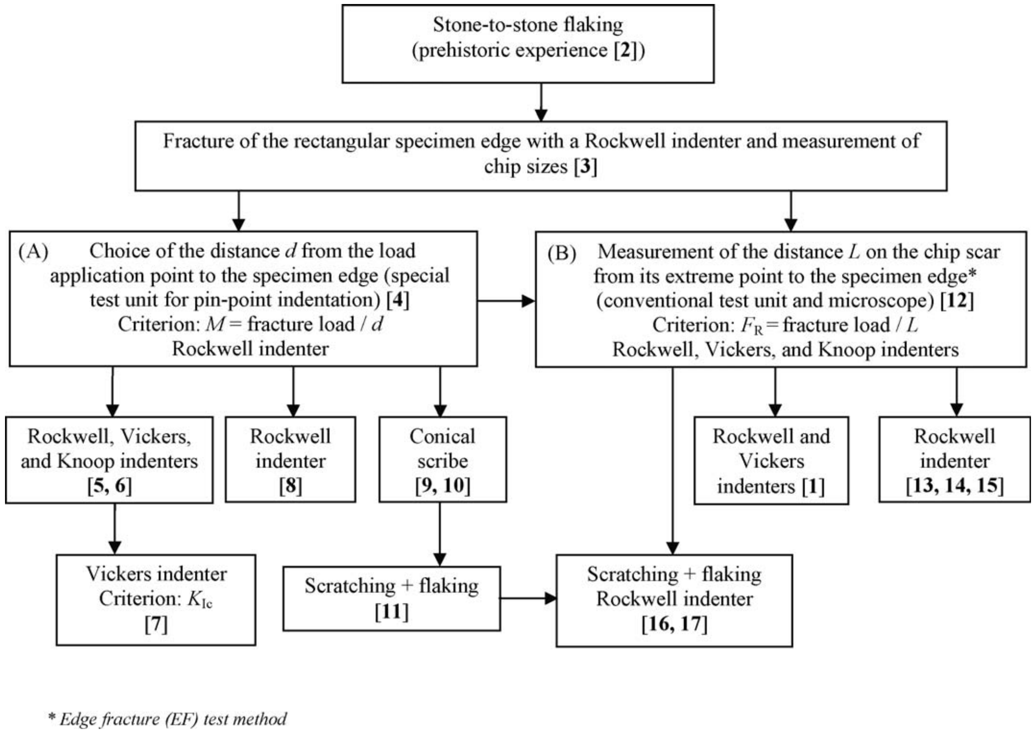

Fig. 1. Flow chart of edge indentation-based fracture resistance tests for brittle materials (see Refs. [2–5,8–11,16]).

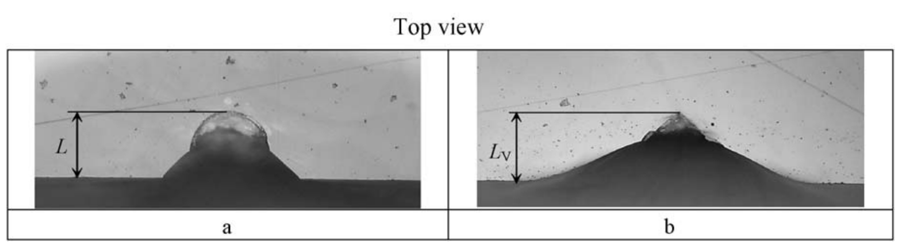

Fig. 2. Measurements of the fracture distances L and L_V on the chip scars of glass specimens (fracture load P_f and L (L_V): 44 N and 0.175 mm (a), 30 N and 0.190 mm (b)).

performed with a CeramTest device mounted on a universal test machine, with a cross-head speed being 0.5 mm/min. In SEVNB tests the loading support was used, for EF tests it was replaced by the indenter holder and *X–Y* table. In EF tests a Rockwell C-Scale standard conical indenter (Gilmore Diamond Tools Inc., USA) with a 200-μm tip radius and a Vickers standard pyramidal diamond indenter were impressed into visually chosen points on the rectangular specimen edge. The fracture resistance of the material was characterized by the ratio F_R (F_{RV}[1]) of the fracture load P_f to the fracture distance L (L_V) from the edge to the extreme point of the chip scar formed on the specimen surface (Fig. 2), which was measured with an Olympus 51MX binocular microscope.

3. Results and discussion

The impression of the conical indenter into the specimen surface first resulted in a Hertzian ring crack, which became the origin for a conical crack that started growing deep into the specimen. This was most evident in glass tests. But because of the edge effect, the chip scar is turned into a quasi-cone[2] (Figs 3a and 4a). The change of loading conditions resulted in the change of chip scar shapes on the specimen edges (Fig. 3b and c), but in Vickers indentations, quasi-cones were revealed only for quartz glass (Fig. 3d). Chip scars on the specimen edges of ceramics most often acquire a scallop-like shape (Fig. 4b).

Experimental results were analyzed using the EF base diagram with the baseline (Fig. 5). As is seen, several results (data points) were located near the baseline, others fell above it, which points to the existence of the barrier to the onset of fracture for the latter. Similar results were also obtained in Ref. [6], where the ratios of the fracture load to the distance from its application point to the specimen edge are equal to 261 N/mm at K_{Ic} = 0.7 MPa $m^{1/2}$ and 318 N/mm at K_{Ic} = 3.0 MPa $m^{1/2}$ for soda lime glass and RBSN, respectively. But this phenomenon was not given proper attention. Though the physical explana-

[1] For Vickers indentation results, the subscript V was introduced.

[2] The term was introduced by Prof. B. Galanov.

Table 1
Properties of examined materials.

Material	K_{Ic} (MPa m$^{1/2}$)	F_R (N/mm)	F_{RV} (N/mm)	F_{Ra} (N/mm)	F'_R (N/mm)	Ψ	Ψ_V	References
Optical quartz glass	0.83 ± 0.02	270	91	79	108	3.40	2.50	State Optical Institute (Russia)
Sc_2O_3	1.49 ± 0.02	143	102	142	141	1.01	1.01	Eastern Institute of Refractory Materials (Russia) [1]
SiC EKasic® F	2.50 ± 0.17	497	163	238	324	2.09	1.53	Wacker-Chemie GmbH (Germany) [12]
SB SiC	2.95 ± 0.02	305	146	281	273	1.09	1.12	Institute for Materials Science Problems (Ukraine) [21]
Al_2O_3-1 (Duralbit 90)	3.00 ± 0.06	300	154	286	281	1.05	1.07	Industrie Bitossi (Italy) [22]
Al_2O_3-2 (submicrometre)	3.43 ± 0.20	519	169	326	342	1.59	1.52	Fraunhofer-Institut für Keramische Technologien und Sinterwerkstoffe (Germany)[a]
Al_2O_3-3	3.87 ± 0.06	340	174	371	357	0.92	0.95	Fothergill Fabrics (UK)
Si_3N_4	4.32 ± 0.12	395	205	411	450	0.96	0.88	[13]
HP Si_3N_4	4.29 ± 0.39	422	180	408	375	1.03	1.13	Institute for Materials Science Problems (Ukraine) [21]
HP SiC	2.45 ± 0.006	483	146	233	273	2.07	1.77	Institute for Materials Science Problems (Ukraine) [21]

[a] Ceramics provided by A. Krell, IKTS, Dresden, Germany.

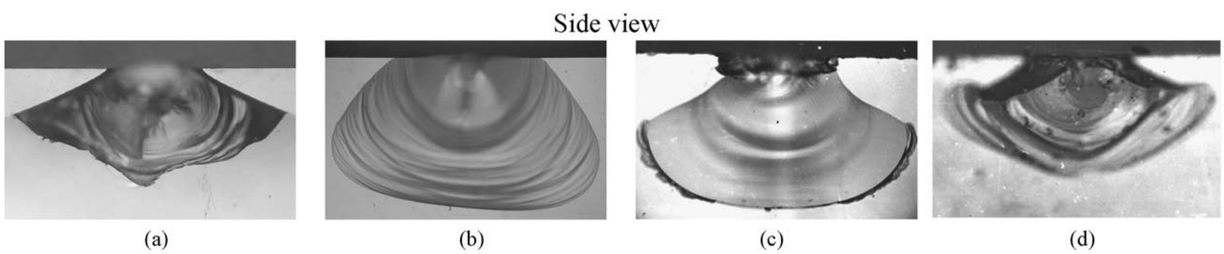

Fig. 3. Chip scars on glass specimen edges formed after Rockwell (a) and Vickers (b) indentations, after scratching and flaking [17] (c) and on an optical quartz glass specimen after Vickers indentation (d) (P_f and L: 40 N and 0.160 mm (a), 27 N and 0.230 mm (b), 17 N and 0.230 mm (c), 10 N and 0.120 mm (d)).

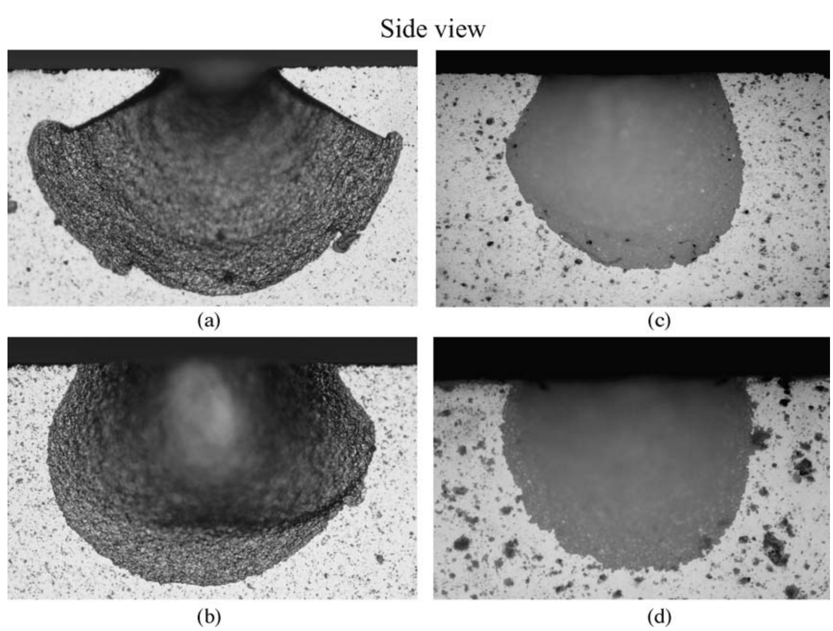

Fig. 4. Chip scars on SiC EKasic® F (a and b) and Al_2O_3-1 (c and d) specimen edges after Rockwell (a and c) and Vickers indentations (b and d) (P_f and L (L_V): 87 N and 0.170 mm (a), 15 N and 0.125 mm (b), 65 N and 0.210 mm (c), 18 N and 0.135 mm (d)).

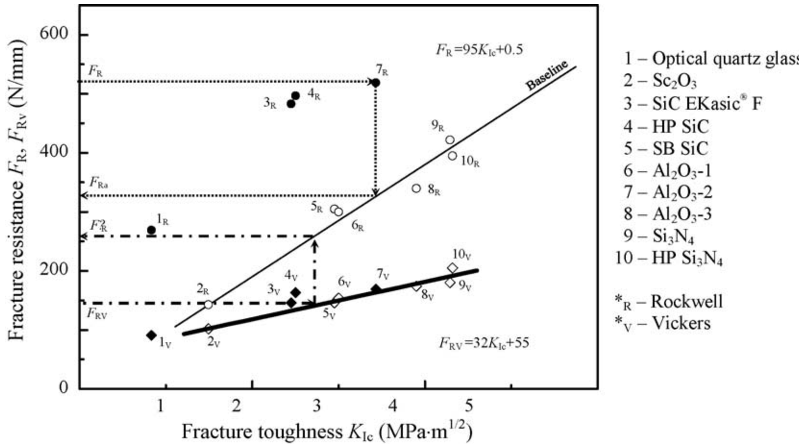

Fig. 5. Base diagram with the baseline [14] and $F_{RV} - K_{Ic}$ relation.

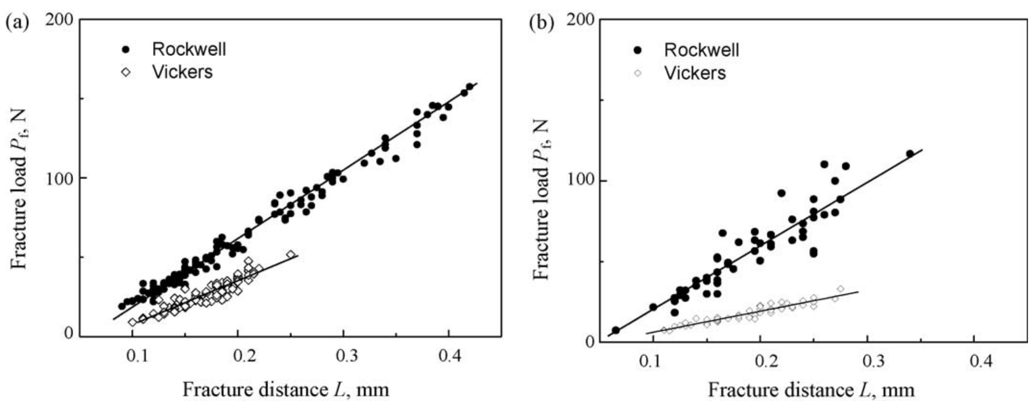

Fig. 6. Rockwell and Vickers $P_f - L$ relations (fracture diagrams) for Al_2O_3-1 (a) and optical quartz glass (b).

tion of this effect is still absent, one may assume that those materials, in contrast to conventional ceramics, possess a higher resistance to the onset of fracture (crack nucleation)[3]. In Rockwell indentations, preceded by the specimen edge scratching (preformation of a stress concentrator), such a barrier was not observed, e.g., for glasses [17]. To estimate the 'level' of this barrier, in Refs. [13,14] additional fractographic investigation of fracture zones on the specimen edges was carried out. In this study, the ratio (damage coefficient k [13]) of the projections of damage zone widths under the indenter to chip scar widths on the side surface of the specimen was determined. It was established that the less this ratio, the higher the barrier to the onset of fracture. The barrier to the onset of fracture can be estimated by the factor ψ, being the ratio of F_R to its apparent value F_{Ra} [15] (pointed lines in Fig. 5). For this estimation not only F_R but also K_{Ic} should be employed, since without the latter the F_R location in the EF base diagram is impossible. Therefore, in both cases for determining the fracture barrier, the two methods should be used, with SEVNB requiring much larger quantities of experimental material, which is not always realizable, especially in materials science research.

A knowledge of barrier levels may be of practical use in studies on ceramics for armour and cutting tools, glasses, etc., since it can become an additional source of information on the ability of the material to resist fracture. Therefore, an effort was made to simplify its estimation. It was taken into account that Vickers indentations (as well as Rockwell tests) demonstrated [1,12] the linear relation[4] between the fracture load P_f and the fracture distance L_V (Fig. 6), i.e. the behaviour of brittle materials changes similarly with the fracture load.

In studies on the barrier to the onset of fracture, the specimen edges were flaked off with both indenters, and the data points were plotted in the EF base diagram (Fig. 5). The data points obtained in Vickers indentations for ceramics, whose behaviour corresponds to the baseline, were approximated with the straight line, described by the equation $F_{RV} = 32K_{Ic} + 55$. It should be noted that the data points for other ceramics obtained in Vickers indentations were located in the vicinity of this line. Since in this case edge flaking started at higher stress concentrations, fracture resistance values decreased and the $F_{RV} - K_{Ic}$ relation, obeying the equation $F_R = 95K_{Ic} + 0.5$,

[3] Transformation-toughened [13,14] and composite [15] ceramics exhibit a lower resistance to the onset of fracture, which in Ref. [24] was considered as a decrease in the energy fracture barrier.

[4] This relation was also confirmed in Ref. [7].

was located at a smaller slope than the baseline in the EF base diagram. Comparative analysis of these relations, with K_{Ic} values being excluded, gave the equation $F'_R = 3(F_{RV} - 55)$, where F'_R is the approximated fracture resistance (relationship between the above characteristics is shown by the dashed line in Fig. 5). Knowing F_R and F'_R values, one can calculate the parameter $\psi_V = (F_R/F'_R)$ (similar to ψ [15]), which can be used to estimate the barrier to the onset of fracture of an examined material. The ψ and ψ_V values are summarized in Table 1. For the materials, whose data points are located near the baseline (insignificant barrier), ψ_V is about unity (~0.85–1.15). But for the materials with a well-pronounced barrier, these values are much higher, e.g., for Al_2O_3-2, SiC EKasic® F, HP SiC, and optical quartz glass, ψ_V is 1.52, 1.53, 1.77, and 2.50. It should be noted that fracture toughness values calculated by $F_{RV} = 32K_{Ic} + 55$, using the baseline (Fig. 5), are somewhat inconsistent (about $\pm$ 10%) with K_{Ic} values obtained by the SEVNB method. This easily realizable and economic procedure may be considered as ''qualitative'' rather than ''quantitative'', being quite sufficient for revealing the barrier to the onset of fracture in an examined material.

4. Conclusions

The behaviour of elastic ceramics and glasses in flaking has been studied. It has been shown that the barrier to the onset of fracture of those brittle materials can be estimated by EF test results using both Rockwell and Vickers indenters.

The results demonstrate the possibility of classifying brittle elastic materials by their resistance to the onset of fracture, which is quite easy to perform in a conventional laboratory, since this procedure requires simple experimental equipment and small material quantities.

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