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Fracture resistance estimation of elastic ceramics in edge flaking: EF baseline

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Abstract

The fracture resistance of single-phase oxide and nonoxide ceramics was studied in flaking the specimen edge with a Rockwell indenter (EF test method) and in bar flexure (SEVNB method). The fracture resistance F_R and fracture toughness K_{Ic} are shown to be proportional, the plot with the F_R-K_{Ic} coordinates is termed the EF base diagram, in which the EF baseline is constructed. It was revealed that the fracture resistance of ceramics was not influenced by chip scar shapes on the specimen edge. The data points for inelastic ceramics with a lower EF barrier to the onset of fracture lay below the EF baseline in the base diagram, while the data points for glasses and ceramics with a higher barrier were located above it.

The EF test method is appropriate for comparative evaluation of fracture resistance of ceramics and verification of estimates obtained by other methods.

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1. Introduction

The fracture behaviour of brittle materials is usually evaluated by conventional methods based on flexure of rectangular bars with a stress concentrator and/or impression of a Vickers indenter into the polished specimen surface. For these methods, the critical stress intensity factor $K_{\rm Ic}$ (fracture toughness¹) serves as the fracture criterion. However, quite reliable information on the ability of brittle materials to resist fracture cannot always be obtained, e.g.,^{2,3} which exerts negative influence on correct fracture resistance estimates. Therefore, a method based on flaking the specimen edge with a Rockwell indenter⁴ deserves further examination. This method is somewhat similar to that used by our remote ancestors in choosing stones for arms and tools.⁵ It is not based on any concepts of the ideally brittle material of linear fracture mechanics¹ or on the model of a crack shape originated in the specimen on indentation.⁶ These models are often greatly simplified as compared to the true material properties.

One of the versions of this method⁷ known as the edge fracture (EF) test method^{8,9,a} provides fracture resistance estimates for conventional elastic ceramics that are proportional to those obtained by the SEVNB method. This relation is known as the *baseline*.⁹ However, the EF fracture behaviour of such ceramics has not been studied extensively enough, though these materials are widely used for different purposes. This situation can cast doubt on the reliability of such a fundamental relation as the baseline, while its practical application opens the way for gaining new information on the behaviour of different brittle materials in fracture. To confirm the above, additional investigations were performed. Their results are outlined in the present communication.

2. Materials and methods

2.1. Ceramics

Linear elastic single-phase isotropic ceramics almost consistent with the concept of the ideally brittle material of linear fracture mechanics¹ were chosen for the investigation. Sintered scandia and yttria (S and Y) from the Eastern Institute of Refractories (Russia),¹¹ Duralbit-90 alumina A-1 (1987) and A-2 (2005) from Industrie Bitossi S.p.A. (Italy), summarized in Table 1, were used for testing. The structure of these ceramics is shown in micrographs (Fig. 1). Hot-pressed silicon nitride HP Si₃N₄⁹ specimens and silicon nitride SN speci-

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^a It was compared with other versions in Ref. 10.

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| Ceramics | Density (g/cm ³) | Elastic modulus E (GPa) | Strength (MPa) | Fracture toughness $K_{\rm Ic}$ (MPa m ^{1/2}) | Fracture resistance $F_{\rm R}$ (N/mm) | |
|----------|------------------------------|-------------------------|----------------|---|--|-----------------------|
| | | | | | All results | $L = 100 - 400 \mu m$ |
| S | 3.79 | 218 | 110 | 1.49 ± 0.04 | $150 \pm 34 (134)$ | $145 \pm 31 (118)$ |
| Y | 4.90 | 193 | 75 | 3.14 ± 0.06 | $336 \pm 65 (105)$ | $329 \pm 63 (95)$ |
| A-1 | 3.49 | 232 | 269 | 2.93 ± 0.08 | $267 \pm 42 (113)$ | $272 \pm 37 (95)$ |
| A-2 | 3.57 | 266 | 329 | 3.01 ± 0.09 | $300 \pm 56 (140)$ | $290 \pm 49 (122)$ |
| SN | 3.13 | 269 | 396 | 4.10 ± 0.06 | $409 \pm 68 (96)$ | $407 \pm 64 (87)$ |
| HPSN | 3.30 | _ | _ | 4.16 ± 0.16 | 417 ± 36 (81) | 418 ± 37 (79) |
| A-999 | 3.86 | 398 | 473 | 3.90 ± 0.16 | $362 \pm 72 (93)$ | $353 \pm 57 (73)$ |

Table 1 Characteristics of examined materials.



Fig. 1. Micrographs of fractured surfaces on S (a), Y (b), A-1 (c), and polished surface of A-2 (d) specimens.

mens, cut out from the cover of the diesel engine cylinder from Toyomenka Kaisha, Ltd. (Japan), were also employed in the experiments. Alumina ceramics A-999,^{12,13} having the structure similar to that shown in Fig. 1d, were used as a reference material.b

As is known, all the above materials are prone to catastrophic fracture after the onset of crack propagation. Since their mechanical behaviour was studied earlier, any unexpected phenomena during experiments were not observed.

2.2. Procedures

The fracture toughness of ceramics was evaluated by the single-edge V-notch beam (SEVNB) method in three-point flexure (20-mm span) of specimens with a $3 \text{ mm} \times 4 \text{ mm cross-}$ section¹² (differs from the standard procedure¹³ only in a smaller span size). For this purpose a CeramTest device (Gobor Ltd., Ukraine) mounted on a universal test machine was used. Its cross-head speed was 0.5 mm/min. The device is equipped with a rigid dynamometer located under the test specimen. It features two steel membranes that ensure precision displacement of the loading rod, as well as a facility on the lower loading support for perpendicular arrangement of the specimen axis relative to the axes of loading rollers.¹⁵

A V-notch was prepared with a special machine. In the specimens, a 200-µm prenotch was cut out with a diamond saw, then a V-notch was polished out with a razor blade distributing a 1-2-µm diamond paste. Its radius did not usually exceed $5-10 \,\mu\text{m}$. The radius and depth were measured on an Olympus

51MX binocular microscope using a Quick PHOTO MICRO 2.3 program.

Specimen fragments formed in those tests were used for further experiments to maintain comparability of results.

Edge flaking tests were performed by the edge fracture (EF) method also with a CeramTest device. But instead of the loading support, the X-Y table with the system of specimen clamping was installed, in its loading rod, indenters were fixed (earlier this technique was employed in studying the fracture behaviour of zirconia crystals¹⁶). Test specimens were glued to photographic glasses clamped on the X-Y table. The indentation point near the specimen edge was chosen with a magnifying glass, after that it was flaked. The flaking was effected with a Rockwell C-Scale standard conical diamond indenter (Gilmore Diamond Tools, Inc., USA). This operation was multiple-repeated for all the specimens (Fig. 2a). The fracture distance L from the extreme point on the chip scar to its edge was measured by an Olympus 51MX microscope (Fig. 2b). These experimental values served for evaluating the fracture resistance of ceramics.

Such a procedure of measuring the fracture distance is a specific feature of the EF test method, making it different from other similar techniques,^{10,17} in which the loading point is chosen with a microscope, thus, the fracture distance is measured from the contact point of the indenter with the specimen to its edge. Fracture loads P_f, which were registered by PC, and corresponding fracture distances L were used to construct $P_{\rm f}$ -L relations (fracture diagrams). The P_f/L ratio was termed the fracture resistance $F_{\mathbf{R}}$.

Since this investigation was aimed at gaining exhaustive information on the fracture behaviour of ceramics, the EF procedure became the basic test method. For these experiments indenters were made to special order, providing precise round-

^b This material was tested in Round Robin,¹⁴ thus, it can be used by its participants to check the reliability of our results.

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