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# Fracture behaviour of Mg-PSZ ceramics: Comparative estimates

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#### Abstract

The mechanical behaviour of different Mg-PSZ ceramics is studied. Results of their edge fracture (EF) and single edge V-notch beam (SEVNB) tests are discussed. These inelastic ceramics exhibit nonlinear relations between the fracture load and the distance from the extreme point on the chip scar to the specimen edge. They also possess nonlinearly rising R-lines. It is established that the data points plotted in the EF base diagram fall below the baseline (lower barrier to the onset of fracture). By projecting these data points onto the baseline, fracture toughness values close to those of the matrix are determined. The limitations of conventional procedures for evaluating the mechanical behaviour of these ceramics were found out. It has been demonstrated that the EF test method can be quite adequate for enhancing the reliability of comparative fracture resistance estimates. © 2009 Published by Elsevier Ltd and Techna Group S.r.l.

Keywords: C. Fracture; Toughness and toughening; D. ZrO<sub>2</sub>; Indentation; Edge flaking (chipping)

### 1. Introduction

For a long time ZrO<sub>2</sub> was used only in the manufacture of refractory ceramics designed for thermal insulation. However, this material had considerable promise, which provided further incentive to studies on its mechanical behaviour. It was established that the ability of cubic zirconia-based refractory ceramics to resist fracture under mechanical and thermal loadings can increase with the content of the monoclinic phase [1]. This effect arises from the introduction of microcracks into the structure of the material, i.e., one of the mechanisms is realized, which fostered development of toughened zirconia ceramics [2]. Interest in structural zirconia ceramics has quickened after a publication devoted to Ceramic steel [3], where it was shown that the stress-induced martensitic transformation of metastable tetragonal particles of this material to a stable monoclinic phase was a mechanism, which absorbs energy and inhibits crack propagation.

Though considerable resources and much effort were spent for the investigation and development of the above ceramics, their use has not become as extensive as first expected. It can also be explained by the fact that their mechanical behaviour was treated almost in the same way as that of the conventional ceramics (or steel) without considering the specific features of this transformation-toughened and inelastic material. All this leads to estimates that are not reliable enough.

The above gave impetus to the investigation, which became the object of the present communication. Basic experiments made use of the SEVNB method [4], built upon the linear fracture mechanics concepts [5], and the edge fracture (EF) test method [6,7] that provides direct estimation (not corrected by any calculation models) of the fracture resistance of brittle materials.

#### 2. Materials and methods

# 2.1. Ceramics

The goal of the present investigation was to gain an understanding of the fracture behaviour of Mg-PSZ ceramics. Therefore, the experiments were based on their representative versions (Table 1), studied earlier. These materials were supplied by ICI Advanced Ceramics (Australia) for making joint studies (comparative tests to check performance of our procedures was carried out on TS-grade ceramics [8]). The specimens of TS and MS ceramics [9], TSE and TSN ceramics, containing 25 and 70% of the monoclinic phase [10] (12% in TS), as well as of SF-S-MS ceramics [6], annealed for another 1 h when compared to normal conditions, were taken as the basic objects of investigation. In addition, SF-S-TS ceramics

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Table 1 Mechanical characteristics of ceramics.

Material	Brittleness measure χ	Elastic modulus (GPa)	Strength (MPa)	Fracture toughness (SEVNB) (MPa m <sup>1/2</sup> )	Fracture toughness (SENB) (MPa m <sup>1/2</sup> )
TS	0.58	198	632	$9.12 \pm 0.14$	$9.67 \pm 0.24$
MS	0.83	187	654	$6.92 \pm 0.26$	$9.18 \pm 0.65$
TSE	0.89	207	563	$4.88 \pm 0.12$	$6.82 \pm 0.27$
TSN	0.65	162	160	$4.03 \pm 0.22$	$4.02 \pm 0.22$
SF-S-MS	0.56	201	600	$9.50 \pm 0.20$	$11.38 \pm 0.26$
SF-S-TS	0.93	193	236	$5.13 \pm 0.14$	$7.18 \pm 0.19$
Y-TZP	1.00	211	774	$5.34 \pm 0.65$	$7.83 \pm 0.67$

were tested, they were annealed for further 12 h in comparison with SF-S-MS ceramics. All these materials were produced from the same powders by similar sintering technology, but their annealing that followed sintering was specific in each case (for theoretical grounds see [9]), which altered their mechanical characteristics, including fracture toughness. Comparative tests were performed on Y-TZP ceramics [11] (Table 1).

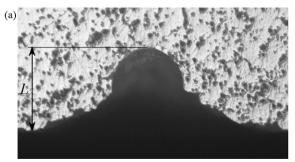
# 2.2. Test procedures

At the first stage of investigations, tests in four-point flexure (20 mm/40 mm span sizes) of  $4 \text{ mm} \times 5 \text{ mm}$  cross-section rectangular specimens were carried out with a CeramTest device (Gobor Ltd., Ukraine), mounted on a universal test machine [12]. As a result, load-deflection curves (deformation diagrams) were plotted and used for determining the strength and static elastic moduli (stress-strain relation covering the initial portion of the deformation diagram) as for estimating inelasticity. The latter is characterized by the brittleness measure  $\chi$  [13], equal to the ratio of the specific elastic energy, accumulated in the specimen to the moment of fracture, to the specific energy, spent for its deformation by the same moment (for elastic ceramics  $\chi = 1$ ). The next step was to study the behaviour of these ceramics under thermal shock loading by a procedure [14]. By this test,  $5 \text{ mm} \times 5 \text{ mm}$  cross-section bars were heated in an electric furnace and then quenched in a water bath at room temperature. After that their residual strength was determined in four-point flexure. The results were presented as furnace-water bath temperature difference  $\Delta T$ -residual strength  $\sigma$  relations (thermal shock resistance diagrams). Then Vickers indentations of polished specimens were performed to analyze the fracture behaviour of these ceramics under local loading. After this, the fracture toughness of ceramics was evaluated by the SEVNB method [4]. In the specimen prepared for the tests, a 200- $\mu$ m prenotch (served as a stress concentrator for SENB tests) was cut out with a diamond saw, and then it was filled with a 1–2- $\mu$ m diamond paste that was distributed with a reciprocating razor, which provided polishing out a V-notch. The V-notch sharpness was measured as a circle diameter inscribed in the notch tip. In these experiments 3 mm  $\times$  4 mm-cross-section ground bars 23–25 mm long were fractured on a CeramTest device with a loading support for three-point flexure. Fracture toughness values, obtained by the SEVNB and SENB procedures, were calculated by the formula usually used for this purpose [15].

Further alternative tests were performed by the EF test method [6,7], when the rectangular specimen edge was flaked off with a Rockwell C-Scale standard conical diamond indenter of a 200- $\mu$ m tip radius (Gilmore Diamond Tools, Inc., USA). The indentation point near the specimen edge was chosen with a magnifying glass, the fracture load  $P_{\rm f}$  being registered by PC. Then the fracture distance L from the extreme point of the chip scar to the specimen edge was measured on an Olympus 51MX binocular microscope (Fig. 1a).

In Rockwell indentations a Hertzian ring crack (primary crack) was first formed near the specimen edge. This is well seen in glass tests (Fig. 1b). From this crack a conical crack started growing deep into the specimen. But due to the distortion of the stress field in this zone, associated with an increase in the material compliance as the indenter is approached the side (open) surface of the specimen, this crack acquired the shape of a quasi-cone, which can be observed in

# View in the indentation direction



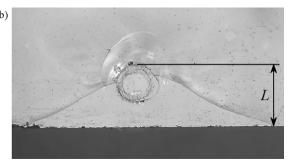


Fig. 1. Chip scar on the specimen edge of TSN ceramics (Nomarski interference) (a) and Hertzian ring crack near the edge of a fused silica specimen (b):  $P_f$  and L for (a) 54 N and 0.22 mm, for (b) 265 N and 0.44 mm, respectively.

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