Available online at www.sciencedirect.com
ScienceDirect
Ceramics International 36 (2010) 345–350
CERAMICS INTERNATIONAL
www.elsevier.com/locate/ceramint

Fracture behaviour of Y-TZP ceramics: New outcomes

G.A. Gogotsi *, V.I. Galenko, S.P. Mudrik, B.I. Ozersky, V.V. Khvorostyany, T.A. Khristevich

Pisarenko Institute for Problems of Strength, 2, Timiryazevskaya Str., 01014 Kiev, Ukraine

Received 10 April 2009; received in revised form 25 June 2009; accepted 15 August 2009
Available online 22 September 2009

Abstract

Modern approaches to fracture resistance estimation of Y-TZP ceramics are critically analyzed. It is shown that fracture toughness test methods do not produce data that are reliable enough. The present investigation makes use of the edge fracture (EF) test method based on flaking the rectangular specimen edge. Chip scars formed on specimen edges after the Rockwell indentation were examined. It is established that not the chip scar shape but its surface area is decisive for the fracture resistance of these ceramics. The influence of indenter sharpness on fracture resistance estimates was elucidated: Rockwell indenter, 400-μm tip radius conical indenter, and Vickers indenter were used. The EF method ensures simultaneous determining a fracture resistance characteristic and plotting an FR-line some equivalent to a conventional R-curve. Therefore, such economically feasible tests, easily realizable in a conventional mechanical laboratory, provide quite exhaustive information on the fracture behaviour of ceramics. The proposed method of evaluating the fracture resistance of Y-TZP ceramics may be useful in materials science practice.

Keywords: C. Fracture; Toughness and toughening; D. ZrO_2; FR-line; Edge flaking (chipping)

1. Introduction

Advanced zirconia Y-TZP ceramics are not only a promising engineering material but also a stock of considerable importance for restoration medicine [1,2]. This fine-grained polycrystalline material, displaying a stress-induced tetragonal–monoclinic (*t–m*) phase transformation, possesses comparatively high strength and fracture resistance, largely controlled by its manufacturing process.

In view of specific fracture behaviour of Y-TZP ceramics, the new ISO standard [3],[1] which stipulates fracture toughness evaluation by the SEVNB method (flexure of a rectangular V-notch beam), does not recommend this method for their testing. This point is extremely important since it actually applies to all other test methods also based on the linear fracture mechanics concepts [4].

It is probably associated with the fact that during Round Robins [5,6] (several results of the latter are summarized in Annex B [3]), estimation of the Y-TZP fracture toughness by different methods did not produce identical experimental data, though the tests were performed in the best mechanical laboratories of the world. The reason (not discussed in [3]) may also be the rising R-curve effect inherent in such ceramics (nonlinear relation between K_{Ic} values and the length of a propagating crack) [7], with this effect the material cannot be uniquely characterized by a single fracture toughness value [4].

The fracture resistance of Y-TZP ceramics is often estimated by the indentation fracture/Vickers indentation fracture (IF/VIF) method based on the indentation of the polished specimen surface and measurement of crack lengths formed near the imprint corners [8]. However, analysis performed for evaluating the validity of this method suggests (confirmed in [9]) that it should not be applied to or be acceptable for any basic fracture resistance measurements of ceramics or any other brittle materials [10]. But these publications do not even allow for the fact that an increase in indentation loads leads to an expansion of *t–m* transformation zone sizes in the subsurface layer of such ceramics.

* Corresponding author. Tel.: +380 44 285 44 64; fax: +380 44 286 16 84.
E-mail address: ggogotsi@ipp.kiev.ua (G.A. Gogotsi).

[1] The SEVNB method is also included in the ISO standard [2] designed for determining service properties of dental ceramics, but Y-TZP is not referred to.

0272-8842/$36.00
doi:10.1016/j.ceramint.2009.09.001

Table 1
Characteristics of zirconia ceramics.

Ceramics	Fracture toughness, K_{Ic} (MPa m$^{1/2}$)	Young's modulus, E (GPa)	Hardness, H (GPa)	Edge toughness (N/mm) E_{tR}	Fracture resistance (N/mm)	
					F_R	F_{Rmax}
Y-TZP-1	3.79 ± 0.14	208	13.57	733 (152)[a]	461 ± 145	619 ± 21 (32)
Y-TZP-2	7.57 ± 0.30	208	13.49	850 (130)	640 ± 130	755 ± 34 (31)
Y-TZP-3	5.34 ± 0.65	211	13.13	742 (124)	570 ± 99	665 ± 26 (32)
TS	9.67 ± 0.14	198	–	707 (175)	483 ± 95	–

[a] Number of chip scars.

Therefore, to avoid erroneous K_{Ic} measurement results, energy absorbed in this process should be taken into account [11]. But despite the above, such methods are still used in practical tests, including biomedical/dental ceramics [12], probably, due to their ease of application and economic feasibility. It is surprising that this important problem, which has long been discussed [11], is ignored by recent review publications [13,14], making them much less comprehensive.

Though the fracture behaviour of Y-TZP ceramics has received much attention, experimental data sufficient to formulate a clear notion of the discussed problem have not been gained as yet. And a test method, providing reliable estimates of the Y-TZP ability to resist fracture, is absent. Such a situation adversely affects optimization of the properties of ceramics and their reliable application. Therefore, it is appropriate to consider an alternative approach to the current problem of fracture resistance evaluation, using a method based on flaking the rectangular specimen edge. Its performance was previously demonstrated on different ceramic materials [15,16]. Considering the above, further Y-TZP investigations have become topical. In the present communication our most recent results are discussed.

2. Materials and methods

2.1. Ceramics

The basic material of this investigation was linear elastic Y-TZP-1 ceramics. Their specimens (3 mm × 4 mm cross-section) were cut from a hollow cylindrical electric heating element manufactured by ICI Advanced Ceramics (Australia). Hot isostatically pressed Y-TZP-2 ceramics (1400 °C in argon, graphite heaters) were used as an additional material [17]. Y-TZP-3 ceramics [6] (Table 1) and inelastic TS zirconia, partially stabilized with magnesia [16], were also examined for comparison.

Taking into account that finishing by grinding and polishing of zirconia specimen surfaces may result in the *t–m* phase transition, a portion of Y-TZP-1 specimens (as in Ref. [11]) was annealed at 1100 °C for 2 h. Since information on Y-TZP-1 ceramics is confidential, the specimens were analyzed to determine their composition (Integrated NMR Chemical Analysis revealed only O, Zr, and Y elements; results of micro-Raman analysis were similar to those obtained for Y-TZP-3 ceramics in Ref. [18]).

2.2. Methods

The edge fracture (EF) test method [19,20],[2] making use of flaking the polished rectangular specimen edge, became the major instrument for the present investigation. The fracture resistance of ceramics was evaluated as the ratio of the load P_f, applied to a Rockwell C-Scale standard diamond indenter (Gilmore Diamond Tools, Inc., USA), to the distance L, measured from the specimen edge to the extreme point of the chip scar on the top surface (Fig. 1). This operation was multiple-repeated, using all the specimens, thus, the fracture resistance of ceramics was characterized by the average ratio designated as F_R. A conical indenter with a 400-μm tip radius, being the modification of a Rockwell indenter, was also used. In this case, the ratio was written as F_{R400}. For Vickers indentations, a diamond indenter (Vilson® Instruments, USA) was employed, this ratio is indicated by F_{RV} [21]. Experimental results were used to plot fracture load P_f – fracture distance L relations (fracture diagrams) and fracture resistance F_R – fracture distance L relations termed fracture resistance (FR) lines.[3] The slope of the straight line approximating the fracture diagram and designated as E_{tR} (edge toughness) can also be considered as the characteristic of the fracture resistance of the material [20] (Table 1).

The tests with a Rockwell indenter, located near the specimen edge and moved over its polished surface to the moment of edge flaking, were also performed. Thus, edge flaking started from a prescratch (additional stress concentrator). As a result, the chip scar formation process changes in comparison with EF tests. For the scratch + edge fracture (S + EF) test method, the fracture resistance was denoted by F_{RS}.

Fracture toughness (K_{Ic}) calculations [22] were based on measurement results obtained in three-point flexure of V-notch rectangular beams (SEVNB method). The notch was polished out with a 1–2-μm diamond paste distributed by a reciprocating razor using a special machine. The V-notch width was measured as the circle diameter inscribed in its tip and did not exceed 10 μm. The K_{Ic} values were calculated by a conventional formula [3].

[2] Its specific features, making it different from other methods based on flaking the specimen edge, are discussed in Ref. [21].
[3] Earlier termed R-lines.