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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

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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.

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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.

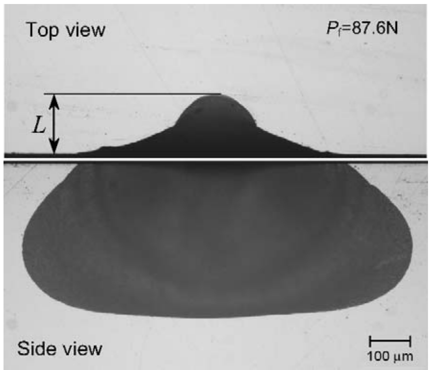

Fig. 1. Chip scar on the specimen edge of Y-TZP-3 ceramics.

The Vickers indentation was used for determining the hardness of Y-TZP ceramics [23].

All the measurements were performed with an Olympus 51MX binocular microscope (50–1000×) using a Quick-PHOTO MICRO 2.3 program.

The experiments were carried out with an independent CeramTest device (Gobor Ltd., Ukraine) mounted on a universal test machine, with a rigid dynamometer located under a test specimen. The speed of the machine cross-head was constant and equal to 0.5 mm/min. For three-point flexure tests, a device was equipped with a loading support to provide the location of specimens perpendicularly to the axis of its loading rollers, they could be given rolling motion when load is applied to the specimen, as indicated in Ref. [3]. In indentation tests the *X–Y* table was mounted over the dynamometer, and indenters were fixed in a moving rod of the test machine. To register loads and time of their action, PC was used, thus, their load–displacement relations could be plotted. These data were necessary for the additional analysis of experimental results.

The S + EF tests were performed on a miniature device of our design, equipped with the *X–Y* table and a block for precision displacement of a Rockwell indenter, which was loaded with calibrated weights [24]. In this case, control is exerted over the indentation load, while in the tests with a CeramTest device the indenter displacement is controlled (soft and rigid loadings).

Specimens for EF and S + EF tests possessed edge radii that did not exceed 10–20 μm, they were given a mirror-like polish, then glued to photographic glasses with Loctite® Super Glue (Henkel Corp., USA) that were clamped on the *X–Y* table.

3. Results and discussion

Annealed and unannealed Y-TZP-1 specimens were first tested by the EF and S + EF methods. It was found that fracture resistance values were almost equal ($F_R = 494 \pm 142$ and 489 ± 155 N/mm, $F_{RS} = 296 \pm 27$ and 320 ± 19 N/mm,

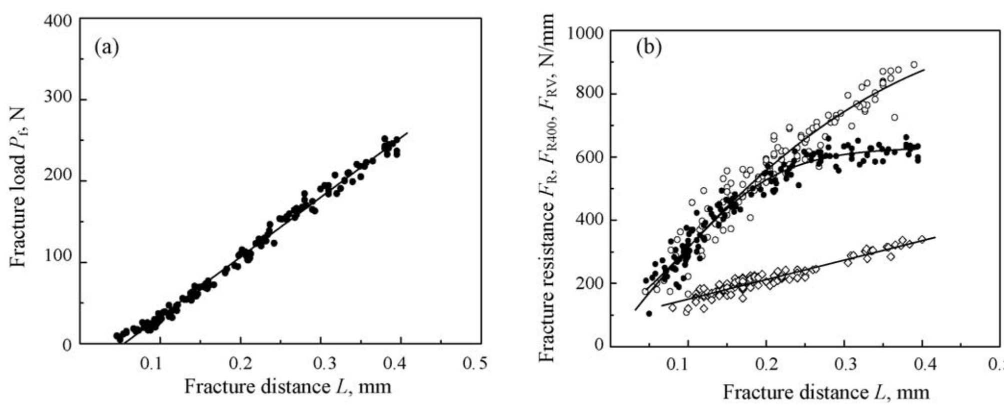

Fig. 2. Fracture diagram (a) and FR-lines (b) for Y-TZP-1 ceramics: (●) Rockwell, (○) conical, and (◇) Vickers indenters.

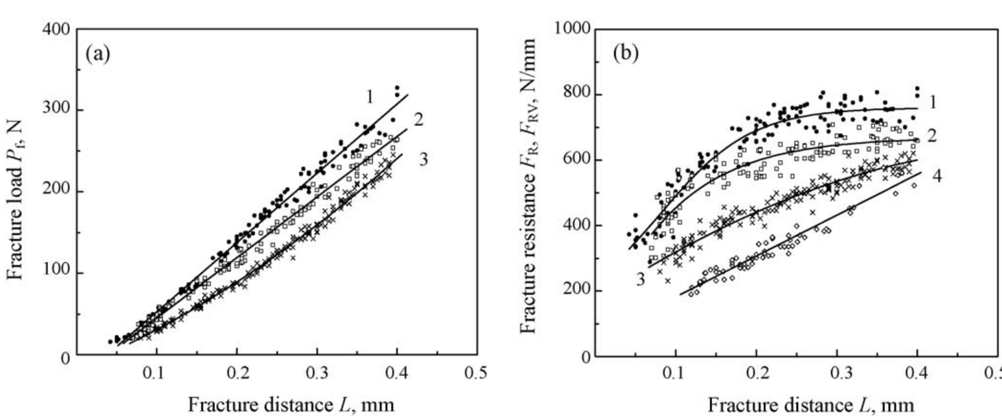

Fig. 3. Fracture diagrams (a) and FR-lines (b) on the Rockwell indentation for Y-TZP-2 (1), Y-TZP-3 (2), and TS (3) specimens, FR-line on the Vickers indentation for TS specimens (4).

respectively). Therefore, to simplify the experimental procedure, unannealed specimens were studied.

The obtained test results are summarized in Table 1, where F_R is the average value for the whole set of experimental data in the range L = 50–400 μm,[4] and F_{Rmax} corresponds plateau on FR-lines (L = 300–400 μm). The fracture diagram and FR-line for Y-TZP-1 ceramics (Rockwell indentation) are presented in Fig. 2, for other ceramics they are shown in Fig. 3. These fracture diagrams (Figs. 2a and 3a) are as linear as those for conventional elastic homogeneous ceramics [19], while for TS (and all inelastic Mg-PSZ ceramics [16]) they are nonlinearly rising. The FR-lines[5] for Y-TZP ceramics (Figs. 2b and 3b) were nonlinearly rising, i.e., similar to those obtained for TS zirconia ceramics and ceramic composites [15].

Thus, in EF tests fracture resistance (F_R) values and FR-lines are determined simultaneously, which is one of the merits of this method.

The data on the fracture behaviour of Y-TZP ceramics (Table 1) created a reliable basis for comparing their fracture resistance (F_R and F_{Rmax}) values with their fracture toughness (K_{Ic}) values. The F_{Rmax} application results in narrower scatter of their values (Table 1), therefore, it can be recommended for use in materials science practice for alternative evaluation of the ability of ceramics to resist fracture.

Comparing FR-lines with R-curves, one should take account of the ability of Y-TZP, as Mg-PSZ [16], to arrest propagating cracks, in contrast to conventional ceramics. The R-curve, being the fracture toughness–crack length relation, can be described as: $K_R(8a) = K_0 + 8K_c(8a)$ [25], where K_0 is the fracture toughness of the matrix (fracture resistance of the material in the transformation zone immediately ahead of the crack tip [26]), $8a$ is the crack length, and $8K_c(8a)$ describes the ability of the material to resist crack propagation (such an approach is examined in Ref [16]). As is seen in Figs. 2b and 3b, nonlinearly rising FR-lines for Y-TZP-1 and TS zirconia ceramics (Rockwell indentation) differ insignificantly, but the second line is smoother, with plateau reached at large L values [17]. It is probably determined by the fact that the process (transformation) zone for Y-TZP ceramics is much smaller than for Mg-PSZ ceramics [27].

When specimen edges were flaked by indenters with different tip radii, the fracture behaviour has changed essentially. In case of Vickers indentations, the results are quite different (Figs. 2b and 3b): FR-lines (F_{RV}–L relations) are nonlinearly rising for Y-TZP and Mg-PSZ as well as differently sloped relative to the axis of the fracture distance L. In other words, the R-curve effect was not revealed. The F_{RS}–L relations obtained in S + EF tests, i.e., when chip cracks develop from an additional stress concentrator (scratch), were straight and almost flat (Fig. 4). When a 400-μm tip radius indenter was used, the effect was rather different (Fig. 3b): FR-

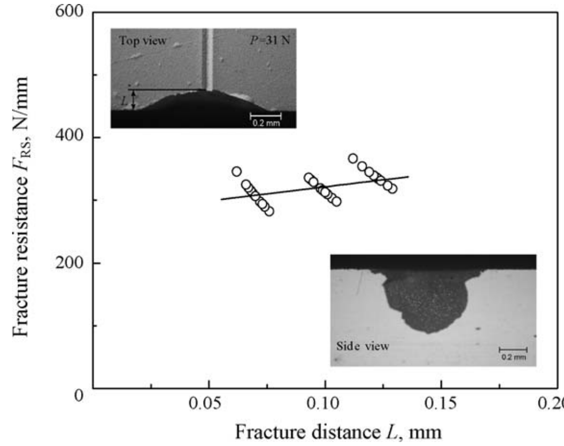

Fig. 4. FR-line for Y-TZP-1 ceramics (S + EF).

lines for Y-TZP ceramics were less curved, similar to those for Mg-PSZ ceramics [16]. The influence of stress concentrations on fracture resistance estimates for ceramics and glasses tested by the EF method was also discussed in Ref. [21]. Investigations in this field are currently going on, and their results will be published.

The analysis of FR-lines also demonstrates that they (as shown for Mg-PSZ ceramics [16]) can be used for the classification of Y-TZP ceramics by their ability to resist crack propagation. These lines (as R-curves) actually correspond to energy spent for fracture of the material [28], therefore, they can be useful as additional information on the fracture resistance of ceramics.

In conclusion, it should be noted that fracture surfaces of chip scars formed on specimen edges in EF tests display much more complex shapes than those formed on fracture surfaces in the tests based on the linear fracture mechanics concepts (corresponding to uniaxial crack propagation). Examining chip scars, formed on specimens edges in the indentation direction, one can divide them into five groups, each corresponding to different FR-line portions (Fig. 5). The primary crack, being a part of a Hertzian ring crack [8], is first formed, followed by two secondary cracks, then the slope of those cracks changes

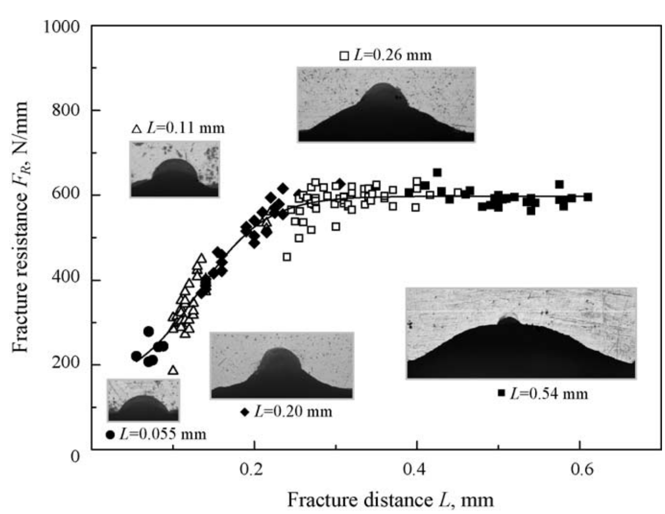

Fig. 5. FR-line and chip scars on annealed Y-TZP-1 specimens.

[4] When several materials are tested at L values over 400 μm, a wide scatter of experimental data is observed, therefore, comparative analysis was performed without overstepping this limit.

[5] FR-lines are shown to be similar [19] to the initial portions of R-curves.

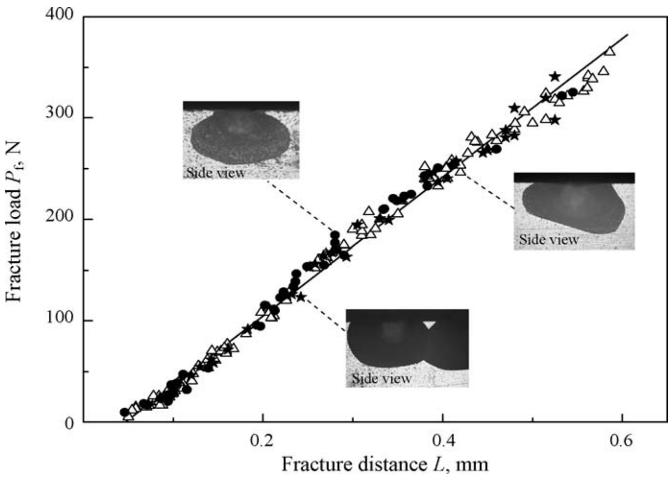

Fig. 6. Fracture diagram for Y-TZP-1 ceramics: (●) symmetrical, (△) asymmetric, and (★) overlapping chip scars, accordingly.

Table 2
Chip scars on Y-TZP-1 specimens.

L (μm)	Fracture resistance, F_R (N/mm)		
	Symmetrical	Asymmetric	Overlapping
50–100	271 ± 46 (11)[a]	265 ± 52 (28)	–
100–140	399 ± 75 (7)	418 ± 68 (29)	431 ± 40 (6)
140–250	585 ± 38 (14)	569 ± 47 (17)	572 ± 37 (9)
250–400	627 ± 10 (9)	614 ± 26 (14)	618 ± 19 (8)
400–600	600 ± 13 (4)	608 ± 17 (29)	608 ± 24 (11)

[a] Number of chip scars.

relative to the specimen edge, and they seem to be concave. With a further increase in indentation loads and as the FR-line plateau is approached, these cracks become convex, with the portions of secondary cracks adjacent to the primary crack located parallel to the specimen edge (such a crack for glass is shown in Ref. [16]). Changes in chip scar shapes with the fracture load P_f are similar for all zirconia ceramics, thus, the fracture process for all tested specimens was similar.

It is well seen that only several ship scars on the side specimen surfaces are symmetrical (Fig. 1), which stems from an imperfect indenter displacement system, microinhomogeneities in the material, and other reasons. This fact may cast some doubt upon the reliability of test results. However, the location of data points in the fracture diagrams (Fig. 6) makes it clear that such a speculation is not valid since all of them are arranged in the vicinity of the approximating line. The data in Table 2, where test results are grouped as in Fig. 5, can lead to the same conclusion. For each typical chip scar, fracture resistance values are very close for loads corresponding to chosen L ranges. Hence, the chip scar area but not its shape controlled the fracture resistance of ceramics, which agrees with earlier results [29].

4. Conclusion

It has been apparent that the test methods for evaluating the fracture resistance of Y-TZP ceramics based on the linear fracture mechanics and indentation fracture mechanics concepts can produce misleading data on the true ability of these materials to resist fracture.

The edge fracture (EF) test method has opened up fresh opportunities for the acquisition of reliable data that can be used without correction by any models. The EF results can characterize the true mechanical behaviour of ceramics under critical service conditions. These outcomes can be considered as the first step in comparative estimation of the ability of Y-TZP ceramics to resist fracture, accounting for the specific features of this process.

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