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Criteria of ceramics fracture (edge chipping and fracture toughness tests)

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Abstract

The methods for evaluating the fracture resistance of ceramic and other brittle materials are discussed. It is shown that the edge-chipping test method may be considered competitive to the conventional methods based on linear elastic fracture mechanics. It allows testing small specimens. This makes it promising for the evaluation of biomedical ceramics and other materials used to manufacture small-sized products or materials from which it is technically difficult or expensive to make ordinary specimens. This energy method differs from the methods based on Griffith's ideas because it evaluates the fracture resistance of materials at all three stages of fracture: crack nucleation, initiation, and propagation. It is confirmed that the data points for ceramics are similar to those of the model material of linear elastic fracture mechanics group along a straight line (called the baseline) on the fracture resistance (F_R) versus fracture toughness (K_{Ic}) diagram (called the base diagram). This is due to the similarity of the fracture surfaces of the chip scars on the edges of specimens. It is shown that the test data of Mg–PSZ and other ceramics can also group along a straight line on the base diagram. It is established that the edge-chipping test methods fail to compare ceramics with dissimilar fracture surfaces of chip scars. The aforesaid is supported by the test data for different ceramic materials.

Keywords: C. Fracture; C. Toughness and toughening; E. Structural and biomedical applications; Indentation

1. Introduction

The problem of evaluating the fracture resistance of brittle materials has attracted attention for a long time: 700–840 thousand years B.C. stones for making tools and weapons were selected by chipping them against each other [1]. In modern phraseology, this is a three-dimensional fracture behavior problem, which is not yet fully theoretically understood [2]. Griffith took the first step toward solving it by addressing a two-dimensional situation and suggested measuring the energy governing the resistance of a material to the formation of new surfaces (energy fracture criterion) [3]. He expressed the stress at fracture as $\sigma = \sqrt{2E\gamma_s/\pi a}$, where γ_s is the surface energy, E is the elastic modulus, and a is the half crack length. Griffith's creative idea contributed to the technological progress because it became possible for the first time to select the most crack-resistant materials (including single crystals [4]) for engineering purposes. Though this criterion opened up the way to the development of fracture mechanics, it was at first ignored. For example, it was not used to evaluate the fracture resistance of the ceramics for the first gas turbines created in Germany during World War II [5].

2. Modern methods for fracture resistance evaluations

2.1. Fracture toughness

In the Bronze and Iron Ages, the suitability of metals was evaluated by testing metallic products. However, the situation changed after metals had come to be used to make highly stressed structures such as bridges, large-capacity vessels, etc., some of which unexpectedly failed [6]. This brought about the problem of evaluating the fracture resistance of metals. At that time Orowan's works appeared useful [7]. They extended Griffith's idea to ductile steel and showed that its fracture resistance is determined by the

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sum of surface energy γ_s and plastic work γ_p (called the effective fracture energy γ_e). In brittle materials, phase transformation and associated microcracking zones at the crack tip, or other effects, may play a role similar to that of the plastic work.

Irwin [8] regarded the ratio of the stored strain energy to the crack length increment as the strain energy release rate G (in this case $\sigma = \sqrt{EG/\pi a}$, where $G = 2(\gamma_s + \gamma_p)$) and introduced the stress intensity factor $K = \sqrt{GE}$ (its critical value K_{Ic} is widely used as a fracture criterion [6]). An important point is that K_{Ic} is dependent not only on the crack-tip stress, but also on the strain energy release rate G_{Ic} during the growth of the crack [6]. These studies laid the foundation for linear elastic fracture mechanics (LEFM), which is the applied mechanics of crack growth, but tells us nothing about specific features of this process [2]. The agreement between fracture resistance evaluations against these two criteria is indicative of their high reliability; therefore, LEFM is the basis for fracture resistance evaluation. Therefore, only the values of K_{Ic} found with the LEFM method can be used to validate other test methods. Unfortunately, this conclusion is often ignored, as in [9], where K_{Ic} measured using the Vickers indentation is considered as a reference. It must be remembered, however, that LEFM-based evaluation will be valid only if the tested material is similar to the model material, i.e., is linear elastic and homogeneous and can be inelastic only near the crack tip [8]. Such are, for example, metals that undergo brittle fracture below the yield point and ordinary elastic fine-grained ceramics. However, this is disregarded by evaluating various brittle materials on the assumption that LEFM methods are universal. As a result, the standards ASTM, CEN, and ISO pass over the specific features in the mechanical behavior of ceramic materials. Because of this, test methods based on LEFM are also applied to determine the fracture resistance of inelastic brittle materials, though elastic–plastic fracture mechanics should be used instead [6]. For example, the J-integral [10] should be used as a fracture criterion instead of the critical stress intensity factor K_{Ic}. It is likely that disregard of the above resulted in the great efforts and costs expended to develop "ceramic steel" [11].

An unfavorable situation also occurs with the K_{Ic} of brittle materials determined by impressing, for example, a pyramidal Vickers indenter into a polished specimen and measuring the size of the cracks at the corners of the indentation (elastic–plastic contact) [12]. It is assumed that these cracks are median (or Palmqvist) ones that, being arrested by internal stresses, do not break the material. The resulting values of K_{Ic} for Ce–TZP ceramics (35 MPa $m^{1/2}$) determined in [13] (and supported in [14]) are greater than the values for metals. Although repeatedly shown to be unfit for the assessment of the fracture resistance of brittle materials [15,16], these methods are often used owing to their appealing simplicity and low material consumption.

2.2. Edge chipping

The National Physical Laboratory (United Kingdom) [17] proposed to determine the fracture resistance of brittle materials by chipping a rectangular edge of specimens with a standard Rockwell indenter (Fig. 1a). In such tests it would be well to consider the surface energy and the fact that this method is somewhat similar to the Hertzian fracture test [12], but the authors chose the easy option of measuring only the fracture load P_f and the distance d from the indentation point to the edge and using the slope of the P_f versus d line as a fracture characteristic. This characteristic, called edge toughness M [18], corresponds to the critical energy release rate G_{Ic} [6] of WC hardmetals, which is confirmed in [19]. But this is not so, however, for non-metallic brittle materials, whose chip scars are different from those on the hardmetals [20]. It was disregarded that dissimilar brittle materials have very differnet chip scars (Fig. 2) and may resist fracture to very different degrees. For instance, in [21], where only the fracture resistance of structural silicon nitride ceramics was studied, there were no problem with the reliability of results. It was also disregarded [18] that the stress and strain distribution

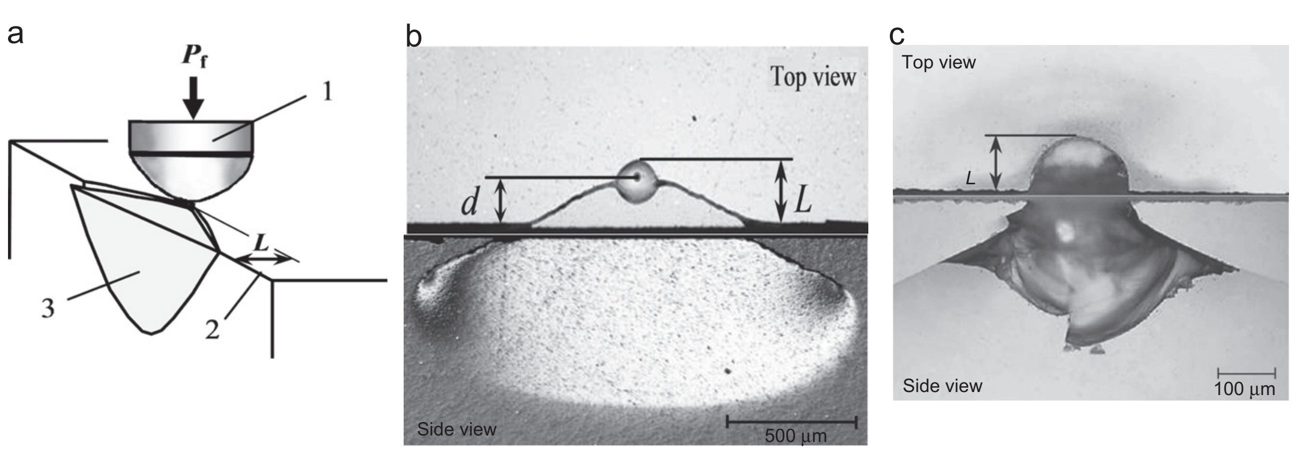

Fig. 1. Schematic of edge-chipping method (a) and chip scars of (Y_2O_3, Al_2O_3)–SN ceramics (c) and quartz glass (b): 1—Rockwell indenter; 2—specimen edge; and 3—chip.

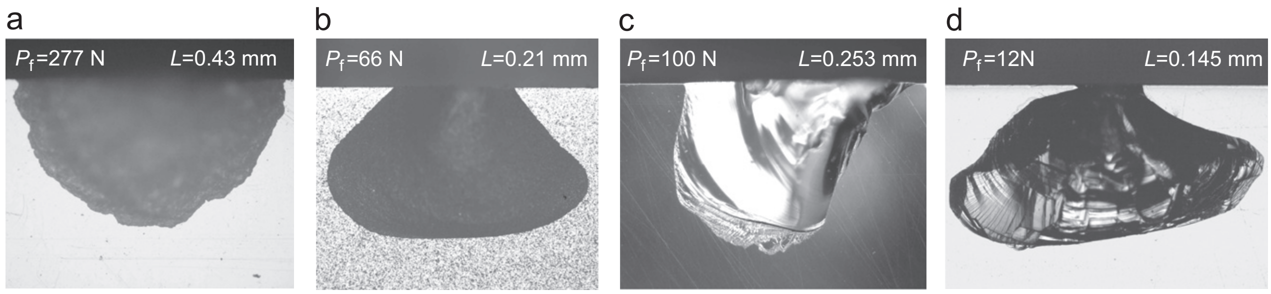

Fig. 2. Chip scars of MS (a) and Duralbit 90 ceramics (b) as well as single sapphire crystal (c) and polycrystalline silicon (d).

at crack initiation [22] is dependent on the distance from the indenter to the specimen's edge (as this distance increases, the point of indentation moves from a quarter-space to a half-space [23],[1]).

3. Nonconventional approach

Our studies were based on [18], but used a test procedure for indentation fracture investigation of brittle materials, including microscopic analysis of fracture zones [26]. This procedure is distinguished from similar ones by measuring the fracture distance not from the point of load application, but from the extreme point of the indentation. This makes the test easier to conduct because no testing machine with a moving microscope is required. After acquisition of statistically reliable test data, a nonconventional approach to the evaluation of the fracture resistance of brittle materials was proposed [24]. The approach involves determining the edge chipping resistance and fracture toughness of the same specimens [27]. The materials were classified according to their deformation behavior [28]. To simplify the explanation of this approach, it makes sense to discuss the tested materials, the test method, and some test results.

The test subjects were inelastic and composite materials similar to but different from the LEFM model solid. All these materials are produced worldwide and were discussed in detail in our previous publications. The test data used here were verified and supplemented (for example, (Y_2O_3, A_2O_3)-SN ceramics [29]). Typical examples of these data are given in Table 1.

The tests were as follows. The rectangular edge of a polished specimen with a rounded radius of less than 15–20 μm was chipped with a standard conical diamond Rockwell indenter C (Gilmore Diamond Tools, Inc., USA). For this purpose a CeramTest device (Gobor Ltd., Ukraine) mounted on a universal test machine was used, which made it possible to record the indenter displacement time as a function of the indentation load. In the test, when this curve had reached its peak, the testing machine was switched off (the indenter speed was 0.5 mm/min). The fracture load P_f was recorded with a computer. The

[1]In [24,25] the optimal distance (100–400 μm) from the edge of the specimen to the extreme point of the chip scar was found experimentally.

Table 1
Materials and tests data.

Material	χ	Fracture toughness K_{Ic} (MPa m$^{1/2}$)	Fracture resistance (N/mm) F_R	F_{Rp}	Number of chips	References
Linear elastic one-phase ceramics						
(Y_2O_3, Al_2O_3)–SN	1	5.50 ± 0.10	542	–	41	[29]
Al_2O_3 (Duralbit 90)	1	3.01 ± 0.10	300	–	140	[20]
SiC EKasic® TM	1	3.60 ± 0.20	509	–	48	Wacker Ceramic AG
TETRABOR®	1	3.10 ± 0.06	417	–	46	
Composite and transformation-toughened ceramics						
Y–TZP-5	1	6.90 ± 0.42	607	743	106	[30]
Y–TZP-3	1	5.34 ± 0.65	570	674	139	[31]
CZ (Al_2O_3/ZrO_2)	1	4.26 ± 0.03	400	518	125	[32]
MS (Mg–PSZ)	0.83	6.92 ± 0.26	455	629	165	[33]
NKKKM-80	0.88	3.03 ± 0.20	147	186	119	[34]
Glass						
Heavy flint	1	0.50 ± 0.01	191	–	122	[35]

distance L (called the fracture distance) from the edge to the extreme point of the chip was measured after tests with a BX51M Olympus (× 50–1000) binocular microscope with QuickPhoto Micro 2.3 software (Fig. 1b, c) The average value of the ratio P_f/L, called fracture resistance, was taken as a characteristic of the material [24]:$F_R = (1/n)\left(\Sigma_{i=1}^{n} P_{fi}/L_i\right)$, where n is the number of chip scars.

An important feature of these tests was an attempt to study all three stages of fracture: crack nucleation, initiation, and propagation. Recall that only two stages of fracture are considered when determining fracture toughness because the first stage is the preparation of a stress concentrator in the test specimen. In these tests, V-notched specimens (SEVNB) were subjected to three-point flexure [27], and K_{Ic} was determined as $K_{Ic} = \left(F/B\sqrt{W}\right)\left(S/W\right)\ \left(3\sqrt{\alpha}/2\right)\cdot Y^*$, where $Y^* = \left(1.99 - \alpha(1-\alpha)\left(2.15 - 3.93\alpha + 2.7\alpha^2\right)\right)/\ ((1+2\alpha)(1-\alpha)^{3/2})$, F is the fracture load, B is the width of the specimen, W is the height of the specimen, S is the distance between the load points, a is the depth of the V-notch, and $\alpha = a/W$ is its relative depth. Special care

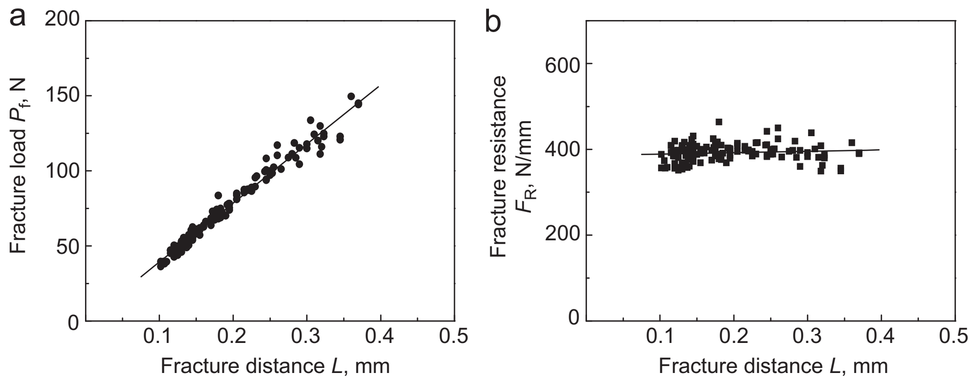

Fig. 3. Fracture diagrams (a) and R-lines (b) of elastic silicon nitride ceramics SN.

was taken to prepare the V-notches (for example, so that the tip radii in Y–TZP ceramic specimens were not greater than 7–10 μm).

Moreover, four-point flexure tests were conducted to record the deflection of the specimen as a function of the load [36]. These data were then used to plot the stress versus relative strains and to calculate the brittleness measure χ (degree of inelasticity) of brittle materials, which is equal to the ratio of stored strain energy to the total strain energy to fracture [37]:$\chi = \sigma_u^2/2E\int_0^{\varepsilon_u} \sigma\, d\varepsilon$,where E is the elastic modulus, σ_u is strength, $d\varepsilon$ is the ultimate bending strain, σ and ε are the current stress and strain, respectively; $\chi=1$ for elastic materials and $\chi<1$ for inelastic materials.

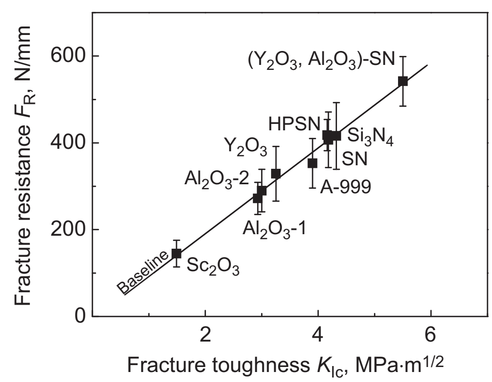

Fig. 4. Base diagram with baseline.

4. Results and analysis

The edge chipping test data were used to determine fracture resistance F_R and to plot P_f versus L (fracture diagrams) and F_R versus L (R-lines) [24], the latter being similar to the initial sections of the ordinary R-curves [6]. Table 1 summarizes the values of F_R and K_{Ic} for typical materials. The fracture resistance of materials that follow a LEFM model is considered first.

4.1. Elastic ceramics

Let us first address ordinary single-phase fine-grained ceramics whose linear fracture diagrams (Fig. 3a) and flat R-lines (as well as R-curves) indicate that these materials are linear elastic and cannot resist the crack propagation because the surface energy is invariant to material properties (Fig. 3b) [6]. Such ceramics exhibit a linear relationship between F_R and K_{Ic}, which was first shown in [24] and supported in [20] (Fig. 4). This suggests the agreement between these two characteristics in the evaluation of the fracture resistance of ceramics (recall the comparison of γ_s, G_{Ic}, and K_{Ic}). It is likely that chip scars on these materials (as well as WC hardmetals [18]) are similar. Thus, the results being discussed may be considered reliable. This kind of relationship is called a baseline [24], and the associated diagram, a base diagram. It is employed in evaluating the edge chipping resistance of brittle materials and validating the values of their fracture toughness. Proving the direct proportionality between F_R and K_{Ic} made it possible to compare different methods of evaluating the fracture resistance of ceramics (Fig. 5) and to position the EF approach among them. It should be noted that, probably, all well-known edge chipping methods produce similar results for materials with similar features in mechanical behavior (see also [38]).

It can be seen from Fig. 6 that the test data points for glass and elastic ceramics used to make armor and cutting tools lie above the baseline [24]. The same data points for inelastic and transformation-toughened materials, however, appear below the baseline (in [28] they were assumed to have a low fracture onset barrier). This is because the fracture resistance of their surface layer is reduced by microcracks, possible phase transformations, and other effects at the beginning of mechanical loading (quasi-plastic materials [39]). The cause of the effect in materials represented by data points above the baseline is different and yet unknown, though it actually controls their serviceableness. It is interesting that Rockwell indentations in materials with such high fracture barrier (Fig. 7a,b) are very different from those on baseline ceramics (Fig. 7c) [24]. The issue of fracture barrier was addressed in studying metals [40], single-crystal silicon [41], and ceramics [42] and in analyzing the first stage of fracture of brittle

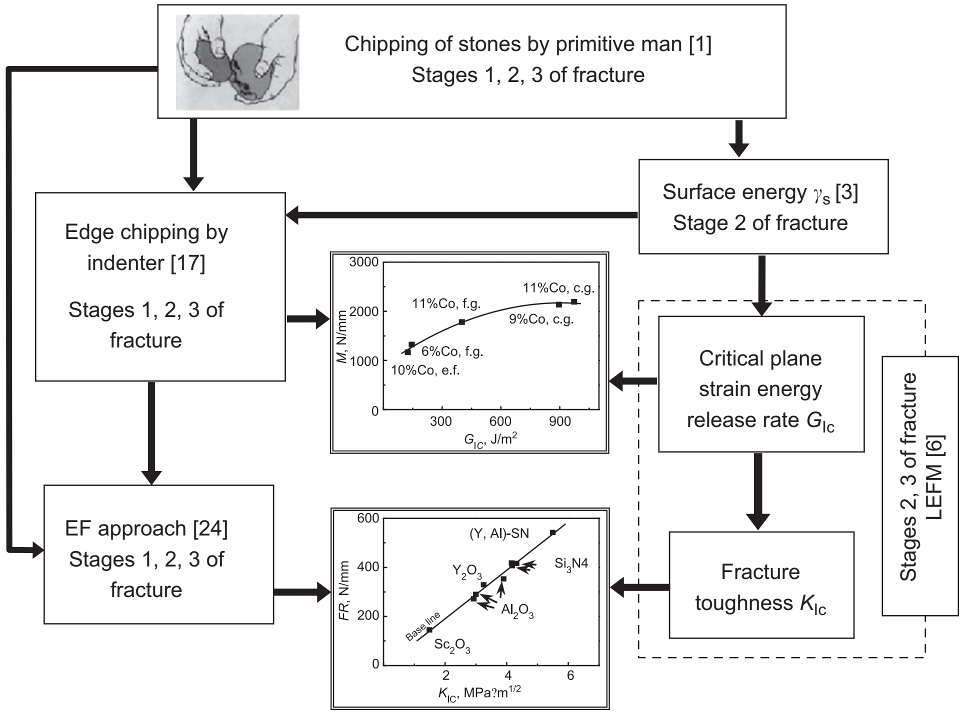

Fig. 5. Relationship between fracture resistance evaluation methods for brittle materials.

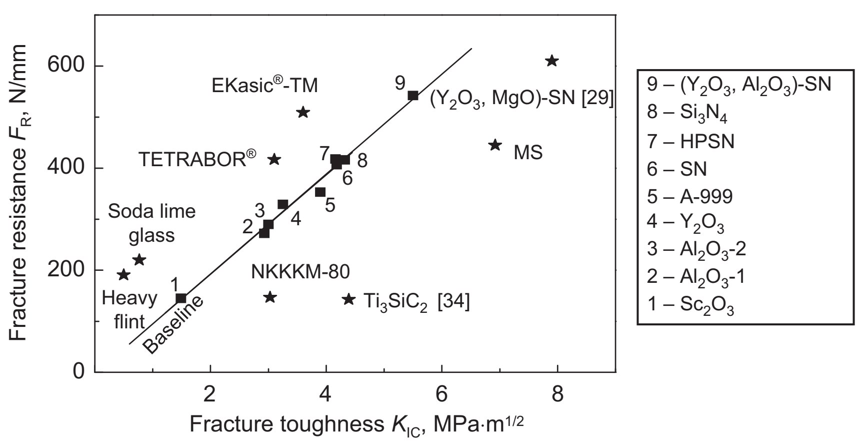

Fig. 6. Base diagram with test data for various ceramics.

materials caused by ball indentation (Hertz problem) [43], but there is not yet clear understanding of this effect. Unless it is explained, it makes sense to regard all ideas related to this effect as debatable. It should also be noted that some materials such as NKKKM-80 and titanium nanolaminate Ti_3SiC_2 (Fig. 6) exert stronger resistance to fracture on the macroscale than on the microscale.

Elastic polycrystalline fine-grained Y-TZP ceramics are characterized by linear fracture diagrams (Fig. 8a). Their fracture behavior is somewhat different from that of ordinary linear elastic ceramics. The nucleation and propagation of a crack in these ceramics is accompanied by a phase transformation that not only absorbs strain energy, but also promotes the formation of a microcracking zone ahead of the crack tip (in this case metals undergo plastic deformation). This causes nonlinear increase in fracture resistance during crack growth (rising *R*-curve) [44]). According to [6], the conventional methods of fracture toughness evaluation may appear unreliable because the section of this curve on which K_{Ic} is determined is a priori unknown. There is a similar situation in edge chipping: *R*-lines are rising (Fig. 8b), which means that the values of F_R of such ceramics may be incorrect. Therefore, for comparison purposes, it might be appropriate to use the values of fracture resistance F_{Rp} on the plateau (linear section) of the *R*-lines, where they are independent of the fracture distance *L*. These data lead to the conclusion that *R*-line behavior (as well as *R*-curve behavior [45]) should be regarded as a basic material property. This seems to be a unique way to reliably determine the difference in the

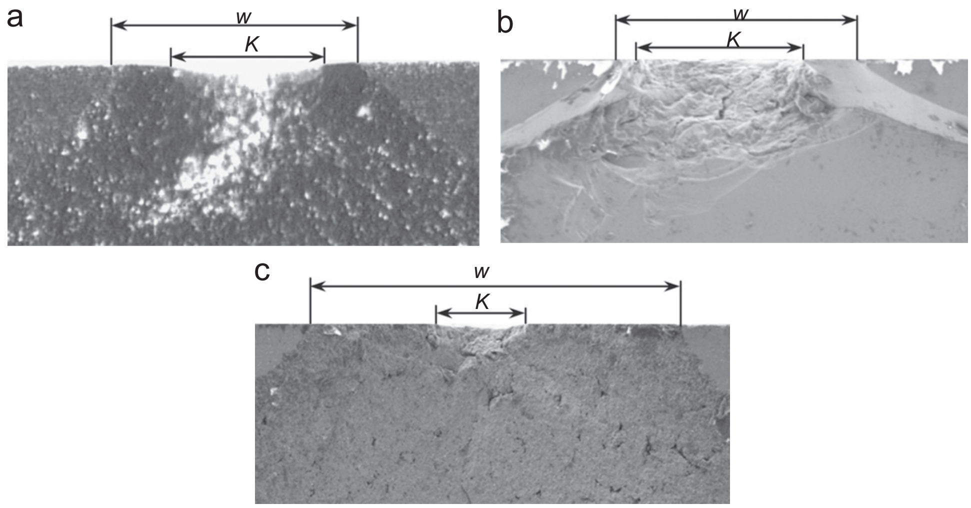

Fig. 7. Chip scars of B_4C ceramics (a) and light crown glass (b) whose data points are above the baseline and Si_3N_4 ceramics with data points on the baseline (d): W is the chip scar size in the indentation direction, and K is the size of the zone damaged by the indenter.

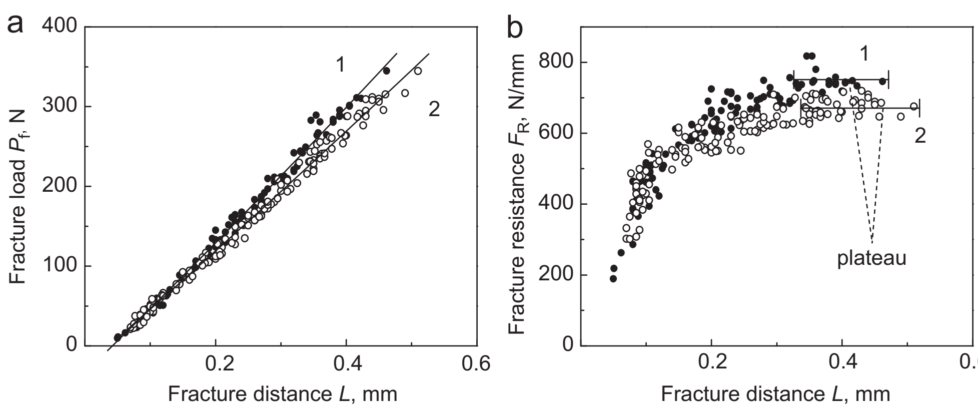

Fig. 8. Fracture diagrams (a) and R-lines (b) of elastic tetragonal zirconia polycrystals: 1—Y–TZP-5 and 2—Y–TZP-3.

fracture resistance of, for example, biomedical Y–TZP ceramics.

4.2. Inelastic ceramics

The mechanical behavior of metallic materials is governed by plastic deformation associated with the motion of dislocations. However, multiphase (including two-phase) ceramics and ceramic compositions can also exhibit inelasticity (brittleness measure χ is less than unity). Among such materials are the above-mentioned transformation-toughened ceramic steel (Mg–PSZ zirconia), composite ceramics with alumina as a matrix and zirconia as a reinforcement, etc. The rising R-curve effect was discovered in studying the fracture of inelastically deformed brittle materials [36]. In [24], this effect was observed in Mg-PSZ ceramics characterized by rising R-lines (Fig. 9b). Note that the same problem was earlier discussed in studying the fracture toughness of similar inelastic zirconia ceramics in [45], where the stress intensity factors corresponding to the plateau of R-curves were analyzed, but the results of these and similar studies have not been put into practice. It can be seen that the R-lines in Fig. 8b do not differ significantly. Therefore, it is legitimate to compare the fracture resistance of such inelastic ceramic materials in the same way as in Y–TZP ceramics, i.e., using values of F_{Rp}. However, their data points lie below the baseline due to a low fracture barrier [24]. It is interesting that their fracture resistance F_R and fracture toughness K_{Ic} are in a linear relationship (straight line in the base diagram, Fig. 10).[2] This is because these ceramics differ only by thermal treatment conditions (their composition is the same), and, probably, the fracture surfaces of their chip scars are similar.

In summary, it should be pointed out that it is reasonable to analyze any edge-chipping test data in the way proposed in [24], i.e., to determine the fracture resistance (F_R) by plotting R-lines (F_R versus L) and using the

[2]Eutectoid-aged 9.9 mol% Mg–PSZ (ICI Advanced Ceramics, Australia).

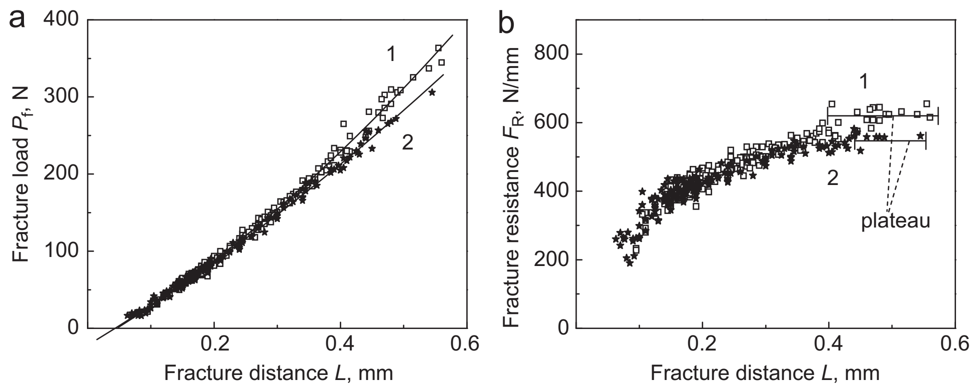

Fig. 9. Fracture diagrams (a) and R-lines (b) of inelastic partially stabilized (Mg–PSZ) zirconia: 1—MS and 2—ASF.

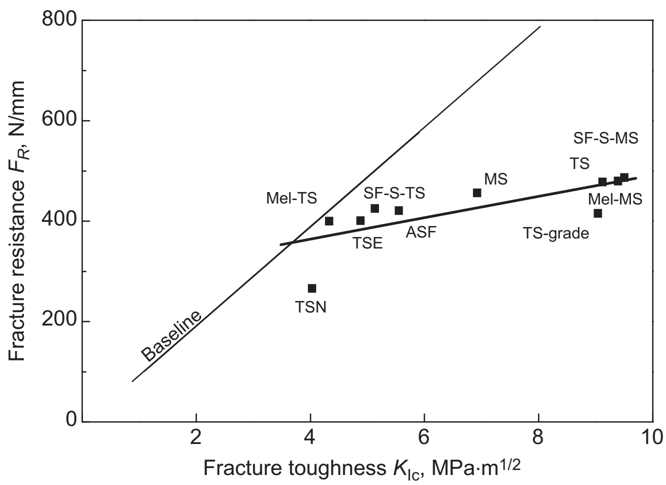

Fig. 10. Test data for Mg–PSZ ceramics shown in base diagram.

baseline and base diagram (F_R versus K_{Ic}). The first step toward this end has already been taken: the standard [46] includes (without reference to [24]) edge chip resistance characteristics $\overline{R}_{eA} = \Sigma_{i=1}^{n} R_{eAi}/n$ that coincide with our fracture resistance characteristic $F_R = (1/n)\left(\Sigma_{i=1}^{n} P_{fi}/L_i\right)$.

4.3. Standardization

The ASTM, CEN, and ISO fracture resistance evaluation standards, which assume flexural fracture of rectangular ceramic beams with different stress concentrators (sharp crack, chevron or V-notch, and Knoop indentation), are based on LEFM and have no fundamental differences from the standard [47] successfully used to evaluate the fracture toughness of metals. In other words, they have all the above-mentioned shortcomings of the ordinary fracture toughness tests. Therefore, repeating the aforesaid, it may be proposed to supplement these standards with an additional method such as an edge-chipping test. This would make it possible to acquire reliable data on the fracture behavior of ceramics whose data points obtained by the two test methods fall onto the baseline.

A different situation occurs with the standardization of methods for the evaluation of the edge-chipping resistance of ceramics. The first step in resolving it is the development of a CEN standard draft [46]. This standard does not distinguish test methods based on edge chipping and on surface scratching followed by edge chipping, wrongly assumes that the fracture patterns on ceramics caused by conical (elastic contact) and pyramidal (elastic–plastic contact) indenters are equivalent [12], and neglects the facts that these materials can be inelastic and exhibit the R-curve effect. It is also disregarded that the standardized method is an energy one and that there is yet no criterion for the correct evaluation of the fracture resistance of various ceramics. Therefore, putting the standard in force would adversely affect the reliability of the evaluation of the fracture resistance of such materials.

5. Conclusion

Based on the results of the present study, it may be concluded that the energy method for evaluating the fracture resistance of ceramics by edge chipping is not yet universal (suitable for comparison of different brittle materials). This is because the reliability of data depends on the chip scar (fracture surface) on the chipped edges. Therefore, it may be used to evaluate the fracture resistance of ceramics that have similar fracture surfaces. Being easy-to-implement and material-saving, this method may be used for ascertaining whether such materials are capable of resisting crack growth (exhibiting R-curve behavior) as well as for materials science research, validation of test data obtained with other methods, and technological comparison of products made of the same material (for instance, under production conditions).

A Round Robin or an inter-laboratory comparison project for assessing the feasibility, advantages, and suitability of edge-chipping test methods would appear to be appropriate. Since there is no general consensus on a unified method (procedure) for the reliable evaluation of the fracture resistance of various brittle materials, it would make sense to hold an international meeting on the subject.

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