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Fracture Resistance of Ceramics: Edge Fracture Method

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Сопротивление керамики разрушению: метод скалывания кромки

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Изучено сопротивление разрушению макрооднородных линейно-упругих керамических материалов из оксидов иттрия, скандия и алюминия, а также из диоксида циркония и нитрида кремния при скалывании кромок прямоугольных образцов индентором Роквелла. Показано совпадение полученных оценок с результатами испытаний на вязкость разрушения, выполненных по методу испытания образцов балочного типа с односторонним V-образным надрезом. При испытаниях измерялась нагрузка, вызывавшая откол части кромки, и расстояние на поверхности образца от этой кромки до места формирования откола. Отношение этих величин рассматривалось как характеристика вязкости откола. Анализ данных, базирующийся более чем на 100 определениях, показал, что они статистически достоверны. Такой метод испытаний, называемый методом скалывания кромки, может использоваться наравне с известными методами определения трещиностойкости керамики. Его применение особенно целесообразно в случае если керамическое изделие по размеру сопоставимо со стандартными образцами или при проведении исследований дорогостоящих материалов (например, нанокерамика).

Ключевые слова: керамика, скалывание кромки, краевая вязкость разрушения, индентирование.

Introduction. Ceramics are widely used in industry, medicine, and everyday life. Being brittle materials, they can undergo catastrophic failure in operation, e.g., even first efforts to manufacture ceramic gas-turbine blades have revealed this critical tendency [1]. An increased sensitivity to stress concentrations and low resistance of the edges to flaking, which can account for uncontrolled failure, also cause certain concern.

With an improvement in the characteristics of ceramics and a continuous growth in production volumes of ceramic items, fracture resistance of these materials attracts the particular attention [2, 3]. In this connection, corresponding national [4], European [5], and international standards [6] are developed as well as comparative evaluation of methods for studying their fracture resistance (Round Robins organized by ESIS, VAMAS, and Japan Society of Fine Ceramics [2]) is performed with the participation of leading specialists of the world. However, in all the cases, ceramics are commonly considered as elastic-deformation materials whose mechanical behavior corresponds to the solid model of linear fracture mechanics [7]. But this is true only for homogeneous ceramics, especially for those materials that do not undergo phase transformations, switching of ferroelastic domains, and other effects changing their crystalline structure and deformation behavior under loading. At the same time, heterogeneous ceramics and different ceramic composites that are deformed inelastically under loading and that do not exhibit catastrophic failure practically escape notice though their production and application grow constantly (in other words, fracture toughness tests do not take account of their mechanical behavior [8]).

The problem can be solved with nonconventional approaches to the evaluation of fracture resistance of ceramics (recall that our remote ancestors choosing stones for weapons and tools, which like ceramics are brittle materials, did not start from any scientific approaches in their practice). Therefore, a study [9] is of interest because it demonstrated that upon flaking off the specimen edge of the brittle material, the load–chip size ratio remained constant irrespective of applied loads. These authors [10] proposed a method to evaluate fracture resistance, according to this method, the specimen edge is flaked off with an indenter in the point chosen with a microscope. The relation between the load P_f flaking off the specimen edge and the distance d from the point of its application to this edge (Fig. 1) was termed the edge toughness M. It was shown to be constant (independent of loads) for tested brittle materials. The edge toughness M – elastic energy release rate $G_{\mathrm{I}c}$ [7] relation was derived for a number of such materials (from glass to superhard alloys) [10] (Fig. 2a). Similar studies on brittle materials (including tool steels) are described elsewhere [11], which confirmed the validity of the $M - G_{\mathrm{I}c}$ relation. But it was noticed that the relation between the edge toughness M and the critical stress intensity factor $K_{\mathrm{I}c}$ was not observed. By convention this test method can be termed the flaking method with a fixed loading point, it was also used by other authors for studying different ceramics [12, 13]. As it was revealed [14, 15], of the materials under study [10, 11], ceramics do not demonstrate an unambiguous relation between M and $G_{\mathrm{I}c}$ values (Fig. 2b).

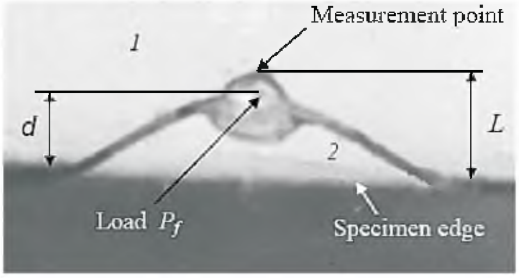

Fig. 1. Surface of the specimen in the indentation direction: (*1*) specimen; (*2*) chip. (Other designations in the text.)

The test method* with an arbitrary loading point [15, 16] also based on findings [9] appeared to be more effective.

It consists in that the indentation point near the rectangular edge of the polished specimen is chosen visually (or by a magnifying glass). The load P_f,

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* Edge fracture (EF) method.

flaking off a part of the edge, is applied to the indenter. Then the fracture distance L (Fig. 1) is measured on the specimen using a microscope (in our case Olympus BX51M, ×1000), this accounts for real fracture patterns of ceramics during their testing. An indentation point is displaced arbitrarily, therefore, the variation of the distance L is of random nature since an exact visual positioning of this point is impossible. The load P_f – distance L relation was used to calculate the flaking toughness E_t.

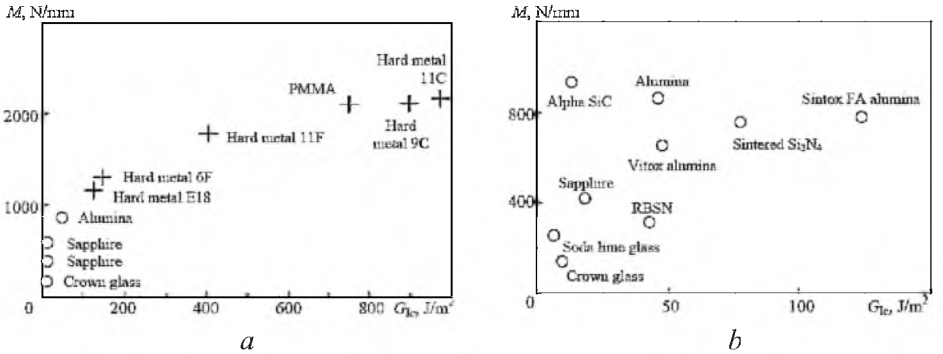

Fig. 2. Edge toughness M – elastic energy release rate $G_{\mathrm{I}c}$ relations for brittle materials (a) and for ceramics (b) [15, 17].

Since the E_{tR} value* for the tested material is constant (Fig. 3), it can be regarded as its mechanical characteristic. It was shown [15] that the flaking toughness – critical stress intensity factor relation appeared to be close to linear for many materials. At the same time, the tests do not require special equipment completed with a microscope.

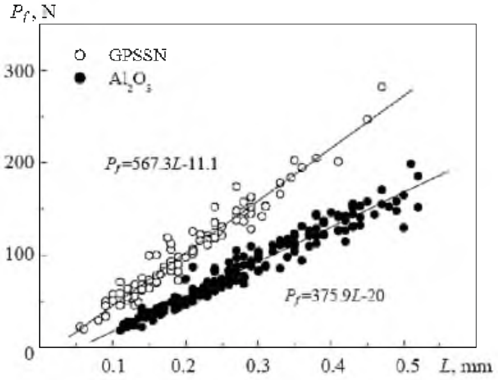

Fig. 3. Fracture diagrams for Al_2O_3-2 and GPSSN ceramics.

Having regard to the importance of the problem and taking into consideration that research [15, 16] (as [10]) was based on a limited number of experimental data, the present investigation, built upon statistically reliable results, is aimed at the development of a standard method for testing fracture resistance of ceramics.

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* The designation of flaking toughness bears an additional symbol R showing that the Rockwell indenter was used in the tests.

Materials and Methods. At the preliminary stage, steps were taken to improve the accuracy of experimental results and to thoroughly choose ceramics for the tests. In particular, the system for fixing the specimen on an $X-Y$ table of a CeramTest universal loading device [2, 15] was modified. In the tests, the Rockwell single-crystal diamond indenter made to special order by Gilmore Diamond Tools Co (USA) was used (indenters of domestic production contained multiple uncontrolled defects). Unlike previous investigations, the present study used only this conical (without ribs) indenter since its use eliminates a rather complicated operation of its orientating relative to the specimen edge.

As earlier [15, 16], the single-edge V-notched beam (SEVNB) method was used to determine the critical stress intensity factor $K_{\mathrm{I}c}$ with the flexure of a V-notched rectangular beam. This method is suitable for testing different ceramics, it provides reliable experimental data based on a limited number of tests [2, 5].

The tests were performed on the specimens in the form of rectangular beams of a 3×4-mm cross-section with an edge radius of about 20 μm whose surfaces were polished with diamond paste after grinding. These specimens were first used for fracture toughness evaluation by the SEVNB method, then on their fragments, flaking toughness by the EF method was determined. All the test results, analyzed below, are statistically reliable, based on the data of more than a 100 experimental determinations.

The prime object of this study is to demonstrate that macrohomogeneous linear elastic ceramics, tested normally by conventional (standard) fracture toughness methods, can show similar results in EF tests.

In the majority of cases, as for Round Robin fracture toughness tests (e.g., [17, 18]), ceramics were industrially prepared, they were linear elastic materials, their mechanical behavior was earlier investigated, which eliminated any unexpected additional effects during testing. Ceramics were different not only in their chemical composition but also in fracture patterns and granularity.

Ceramics of primary emphasis were produced on the basis of alumina (Al_2O_3) in Italy, zirconia (Y-PSZ) in Ukraine, yttria and scandia (Y_2O_3 and Sc_2O_3) in Russia, silicon nitride (GPSSN) in Switzerland and (Si_3N_4) in Japan. Australian Mg-PSZ ceramics were an additional object of investigation (Table 1).

Results and Analysis. Test results (as in other similar studies) were used to construct fracture diagrams ($P_f - L$ relations) with linear regression approximation of experimental data, flaking toughness (E_{tR}) values were calculated from the equation $P_f = E_{tR}L + \Delta P$, where ΔP is the magnitude of approximating line displacement along the P_f axis (Fig. 3). As it was mentioned above, flaking toughness can be the ratio of the load P_f to the fracture distance L. Thus, the arithmetical mean of this ratio was also determined for each material. In this case, to exclude possible confusion, flaking toughness was designated as FT. It should be noted that experimental data, used in the analysis and summarized in Table 2, demonstrate a certain difference in flaking toughness values with different approaches to their calculations.

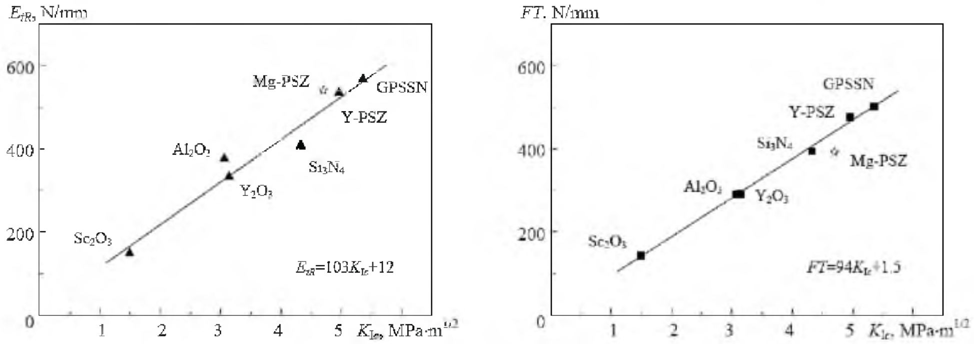
The test results became the basis for plotting the $E_{tR} - K_{\mathrm{I}c}$ relation (Fig. 4a), which exhibits a certain scatter in data (as a similar diagram [16]). To clarify this phenomenon, the correspondence of data distribation to the normal law was examined, the latter being the basis for the linear regression analysis [20] used in

E_{tR} calculations. The statistical test revealed (Table 3) that in many cases, experimental data obtained in EF tests did not correspond to the normal distribution of random values (Fig. 5), which was not given proper attention. Therefore, the data (Table 2) were also used to construct the $FT - K_{\mathrm{I}c}$ diagram (Fig. 4b). The Figure demonstrates that this diagram, as the $E_{tR} - K_{\mathrm{I}c}$ diagram, is linear but exhibits a somewhat smaller scatter in data. The analysis confirms that ceramics with zero fracture toughness possess practically zero flaking toughness, which is indicative of strong grounds to employ the EF method along with conventional ones for fracture toughness testing of ceramics. Moreover, it is more appropriate to calculate flaking toughness as the ratio of the load P_f to the fracture distance L. The $FT - K_{\mathrm{I}c}$ diagram can also be considered the basis for comparative evaluation of the fracture resistance of linear elastic ceramics, which is of interest, especially in those cases when it is impossible (or inappropriate) to prepare standard specimens for conventional fracture toughness tests because of a limited quantity of a material or its high cost.

Table 1

Characteristics of Ceramics

Ceramics	Nomen-clature	Density ρ, g/cm^3	Brittleness measure χ	Elastic modulus E, GPa	Strength σ, MPa	References
Alumina	Al_2O_3-1	3.70	1.0	232	322	–
Zirconia	Y-PSZ	6.05	1.0	197	425	[2, 3, 15]
Yttria	Y_2O_3	4.77	1.0	155	60	[19]
Silicon nitride	GPSSN	3.23	1.0	320	920	[2, 3, 15, 17]
Silicon nitride	Si_3N_4	3.16	1.0	273	468	–
Alumina	Al_2O_3-2	3.49	1.0	322	269	–
Zirconia	Mg-PSZ	5.62	0.8	183	550	–
Scandia	Sc_2O_3	3.79	1.0	218	110	[19]



a *b*

Fig. 4. Flaking toughness E_{tR} – load P_f (*a*) and $FT - K_{\mathrm{I}c}$ (*b*) relations for the ceramics under study.

Table 2

Experimental Results

Nomenclature	K_{Ic}, MPa · m$^{1/2}$	Number of chips	Flaking toughness		
			Linear approximation		FT, N/mm
			E_{tR}, N/mm	ΔP, N	
Al_2O_3-1	3.06 ± 0.15	138	370.5	−18	308.6 ± 38.6
Y-PSZ	4.96 ± 0.18	125	533.3	−9.3	475.3 ± 47.2
Y_2O_3	3.14 ± 0.06	113	332.3	−5.7	291.5 ± 55.0
GPSSN	5.36 ± 0.34	100	567.3	−11.1	501.7 ± 66.5
Si_3N_4	4.32 ± 0.12	154	404.4	−1.6	394.2 ± 29.4
Al_2O_3-2	2.93 ± 0.08	140	328.8	−10.5	266.0 ± 43.1
Mg-PSZ	4.70 ± 0.40	158	540.8	−45.6	392.0 ± 63.2
Sc_2O_3	1.49 ± 0.02	71	160.1	−2.3	145.5 ± 31.8

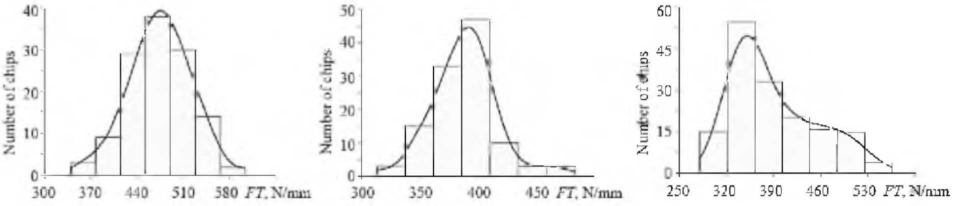
Note: ± is the standard deviation.

Table 3

Statistical Test of Chip Size Distributions on Ceramic Specimens

Nomenclature	Normality criteria					
	Shapiro–Wilk	Anderson–Darling	Martinez–Iglewicz	Kolmogorov–Smirnov	D'Agostino	
					skewness	kurtosis
Al_2O_3-1	+	+	+	+	+	+
Y-PSZ	+	+	+	+	+	+
Y_2O_3	+	+	+	+	+	+
GPSSN	+	–	–	+	+	+
Si_3N_4	–	–	–	–	+	–
Al_2O_3-2	–	–	–	+	–	+
Mg-PSZ	–	–	–	–	–	+
Sc_2O_3	–	–	–	–	–	+

Note: (+) accept, (–) reject.



a *b* *c*

Fig. 5. Histograms based on statistical data analysis for different ceramics: (*a*) Y-PSZ; (*b*) Si_3N_4; (*c*) Mg-PSZ.

Fracture diagrams are worthy of notice because they clearly demonstrate the scatter in experimental data and the behavior of the material upon fracture. These diagrams, being linear for linear elastic ceramics, appeared to be nonlinear (e.g., Fig. 6) for inelastic ceramics. It is probably associated with the fact that an increase in the chip scar surface (fracture surface characterized by the fracture distance L) caused an increase in the fracture resistance of these ceramics (E_{tR} in Table 2 corresponds to linear data approximation). For example, if the diagram (Fig. 6) is divided into 0.1–0.3, 0.30–0.45, and 0.45–0.60-mm portions, the E_{tR} values are equal to 410, 601, and 821 N/mm, respectively. When the calculations employ tangents to this diagram, the curve similar to the R-curve can be derived [7]. By this is meant that the fracture toughness evaluation for such ceramics by conventional test methods is not legitimate (it also refers to FT values). It should be noted that similar results are obtained by the analysis of R-curves, which is quite a complicated experimental problem. In connection with the above, the test results for these ceramics are not included in the diagram (Fig. 6).

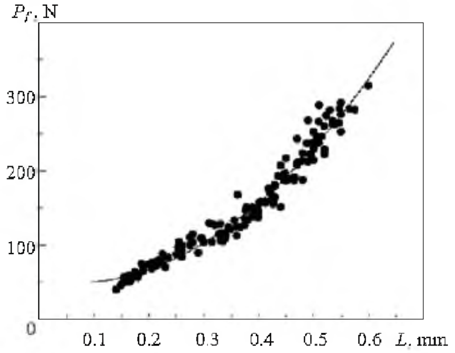


Fig. 6. Fracture diagram for Mg-PSZ ceramics.

Finalizing the analysis of results, it should be remarked that chip scars, formed on the same specimens during their testing by the same procedure, are not always “expected” (symmetrical or similar to each other), their shape is not always the same for different ceramics. This issue would be studied thoroughly in further investigations devoted to this problem.

Conclusions. The results of this study show that fracture resistance determinations by the EF method for the ceramics under study are proportional to critical stress intensity factor values obtained by the SEVNB method. This confirms the earlier conclusion that flaking toughness can be considered as a fracture toughness characteristic of ceramics. The investigation corroborates the performance and potentials of a fundamentally new method for studying the fracture resistance of ceramics (EF method).

Резюме

Вивчено опір руйнуванню макрооднорідних пружно-лінійних керамічних матеріалів з оксидів ітрію, скандію й алюмінію, а також із діоксиду цирконію та нітриду кремнію при сколюванні кромок прямокутних зразків індентором Роквелла. Показано, що отримані оцінки збігаються з результатами випробувань на в'язкість руйнування, які проводяться за допомогою методу випробування зразків балочного типу з одностороннім V-подібним надрізом. При випробуваннях вимірювалося навантаження, яке призводило до відколу частини кромки, і відстань на поверхні зразка від цієї кромки до місця формування відколу. Відношення цих величин розглядалося як характеристика в'язкості відколу. Аналіз даних, що базуються більш як на 100 визначеннях, свідчить про їх статистичну достовірність. Такий метод випробувань, названий методом сколювання кромок, може використовуватися нарівні з відомими методами визначення тріщиностійкості кераміки. Його використання особливо доцільно у випадку, якщо виріб із кераміки за розмірами можна порівняти зі стандартними зразками, або при проведенні досліджень дорогих матеріалів (наприклад, нанокераміка).

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