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Fracture toughness of ceramics and ceramic composites

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Abstract

The fracture toughness of zirconia, alumina, and silicon nitride ceramics, zirconia and alumina single crystals, silicon carbide as well as silicon nitride ceramic particulate composites, silicon nitride laminated composites, and other ceramics materials were studied by a single edge V-notched beam (SEVNB) method. Manual and mechanical procedures for V-notch polishing-out and three- and four-point flexure tests were developed. Load–deflection diagrams for V-notched specimens contributed to better understanding of the deformation behavior of ceramics at room temperature and 1300–1400 °C. SENB (single edge notched beam) and SEPB (single edge precracked beam) as well as micro-Raman spectroscopy data were used to analyze the SEVPB results. The ratio between SEVNB and SENB results is about 0.6 for elastic materials, over 0.9 for inelastic ones, and about 1.0 for laminated ceramic composite. The polishing-out of a V-notch does not lead to the tetragonal-monoclinic phase transformation in zirconia ceramics.

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1. Introduction

Ceramics and ceramic composites are promising materials having rather high strength characteristics but quite low crack resistance properties at the same time. This is one of the major factors hindering the wide-scale application of these materials in various fields of human activities. The crack resistance is critical not only for ceramic products operating under extreme mechanical and thermal loads but also for structural components whose brittle fracture is intolerable even under arbitrary loads.

For many years, the performance of ceramics has been evaluated on the basis of full-scale tests. However, their fracture toughness characteristics have not always been the object of scientific interest. Wide-scale fracture toughness investigations were started only in the late 1980s. International prestandard studies (Round Robin on Fracture Toughness–RRFT [1]) aimed at the assessment of the accuracy and reliability of the data obtained by commonly accepted test methods were important

steps in this field. Leading specialists from different countries engaged in those investigations performed statistical analyses to compare the test methods summarized in Table 1. They were helpful for creating the basis of more reliable determination of the fracture toughness characteristics of ceramics. For a long time, no particular preference was given to any of the methods considered because each of them had appreciable shortcomings, which restricted their application [2,3]. Recently, much attention has been focused on the SEVNB method [4], which is a further development of a simple and widely used the single edge notched beam (SENB) method suitable only for rough fracture toughness evaluations. The test data obtained by the SEVNB method were found to be comparable to those obtained by the single edge precracked beam (SEPB) method usually considered as providing the real values of critical stress intensity factor (K_{Ic}) but this test is often difficult to realize in practice [5]. To evaluate the potentials of the SEVNB method as a standard one for testing ceramics, its analysis was performed [6]. We verified the efficiency of our procedures and demonstrated the applicability of the SEVNB method to different ceramic materials.

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2. Experimental

2.1. Materials

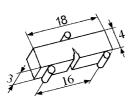
Isostatically pressed silicon nitride ceramics (Si₃N₄) [7], sintered (Y-PSZ) [8] and sintered under pressure (Y-PSZa) partially-stabilized zirconia ceramics [9] were the major materials used in the process of verifying the experimental procedures. Gas-pressure sintered silicon nitride (GPSSN) as well as sintered alumina (Al₂O₃-998) and silicon carbide (SSiC) [6] analyzed in the RRFT'97 also became the objects of the investigation. In our experiments we also used zirconia and alumina single crystals as well as silicon nitride based laminated composites with compressed Si₃N₄ and tensile-stressed $Si_3N_4 + n$ wt.% TiN layers [10]. This composite, layers of which were prepared by rolling, was produced by hot pressing at 1850 °C for 20 min under pressure 150 MPa. For high-temperature investigations, reaction-bonded and sintered $Si_3N_4 + 30\%$ SiC + 3% MgO [11] and hot-pressed (in graphite molds) SiC + 50%ZrB₂ + 10% B₄C [12] ceramic particulate composites were also used. Some data on these ceramics are given in Table 2.

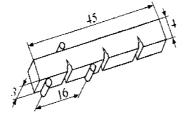
2.2. Test specimens

Rectangular beams (3 mm×4 mm cross-section) Vnotched in two stages were used as test specimens [7]. The package of 2-6 specimens were glued onto a ceramic plate and then V-notched. Two additional specimens were fixed on both sides of the package for its protection from the edge effects due to notching. A preliminary notch 0.8 mm deep (specimens for fourpoint flexure) or 1.6 mm deep (specimens for three-point flexure) was cut in the package by a 150-300 µm thick diamond disk. At the next stage, the V-notch was manually (convenient for comparatively "soft" ceramics) or mechanically (necessary for "hard" ceramics) polished-out after filling the preliminary notch with a diamond paste of grain sizes ranging from 2 to 7 µm and a razor blade 150-300 µm thick. In the case of manual polishing-out, the walls of the preliminary notch (1.6 mm deep) served as the razor blade guides. The purpose of the test determined the number of notches (one or three) cut in a standard 45-mm long specimen (Fig. 1). The criterion for the proper V-notch sharpness is its width, S, equal to two root radii ρ . The notch

Table 1 Conventional ceramics fracture toughness test methods

Specimen	Method and fracture surface of specimen	Specimen	Method and fracture surface of specimen
P. S. W.	SEPB (single edge precracked beam)		SCF (surface crack in flexure)
P S S	SENB (single edge notched beam)	De div	IS (indentation strength)
	CNB (chevron notched beam)		IF (indentation fracture)





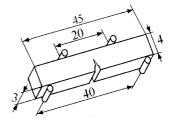


Fig. 1. SEVNB specimens: a, b, three-point flexure; c, four-point flexure.

length and radius were measured with optical (Olimpus BX51M) and scanning electron microscopes (×500 or better).

2.3. Procedures

The specimens were tested in three-point (16 mm span between the bearing rollers) or four-point flexure (20–40 mm spans between the bearing rollers) on a home-made Ceramtest block, mounted on a universal testing machine and fitted with a hard load cell, a system for precision displacement of a loading rod, and loading supports. The latter are equipped with an attachment for the rotation of the bearing rollers during specimen loading. Three-point flexure required the precise alignment of a specimen on the bearing rollers, with the axis of the central bearing roller and the radius of the V-notch root being in the same plane as the applied load. In high-temperature tests, we used a loading block which is similar to that used in room temperature tests.

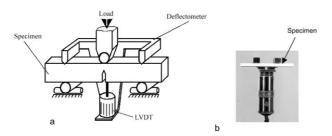


Fig. 2. Loading scheme for three-point flexure (a) and deflectometer suspended on a specimen $3\times4\times45$ mm³ (b).

Table 2 Characteristics of some materials For the recording of the load-deflection curves, a high-sensitivity LVDT-based deflectometer was suspended on a specimen (Fig. 2) and was in no way connected to the testing arrangement [13]. The LVDT was located outside the heating chamber in case of high-temperature tests.

In all the experiments, the speed of the testing machine crosshead was constant and equal to 0.5 mm/min. Load cell and deflectometer readings were recorded with a coordinate potentiometer.

Fracture toughness, $K_{\rm Ic}$ values were calculated in accordance with ASTM standard [14] (three-point flexure tests) and DIN one [15] (four-point flexure tests). Additional procedures for evaluating $K_{\rm Ic}$ and other mechanical characteristics are described elsewhere [16].

A micro-Raman imaging microscope (Renishaw, USA) with a 514.5 nm excitation line of an argon laser ($\times 100$ objective and a ~ 1 - μ m diameter spot) was used to examine the surfaces of fractured Y-PSZ specimens by a procedure described in Ref. [17].

3. Results and discussion

The results of comparative three- and four-point flexure tests of monolithic ceramics and particular ceramic composites are summarized in Table 3, where the data obtained within the RRFT'97 program are also cited. The variation of $K_{\rm Ic}$ values as a function of notch root radius was studied for silicon nitride and zirconia (Fig. 3). Load–deflection diagrams for V-notched specimens are

Material	Density (g/cm ³)	Strength at 20 °C (MPa)	Hardness (GPa)	Method of manufacture
Si ₃ N ₄	3.14	700	14.1	HIP
GPSSN	3.23	> 920	13.5	Gas-pressure sintered ^a
SSiC	3.15	_	22.4	Sintered ^a
Al ₂ O ₃ -998	3.86	350	19.3	Sintered ^a
Y-PSZ	6.05	425	12.1	IP + sintered

^a Material of RRTF'97.

Table 3 Comparative fracture toughness tests (MPa $\rm m^{1/2}$)

Test method	Three-point flexure (a/W≈0.5)	Four-point flexure (a/W≈0.20.3)		
		Our results	RRFT'97 results	
Si ₃ N ₄	5.5±0.07 (5) ^a	5.35±0.16 (5)	_	
GPSSN	5.3 ± 0.04 (5)	5.2 ± 0.18 (5)	5.36 ± 0.34 (129)	
$Si_3N_4 + 30\% SiC + 3\% MgO$	2.27 ± 0.14 (4)	2.40 ± 0.16 (5)	_ ` ` `	
SSiC	2.45 (1)	2.66 ± 0.20 (4)	2.61 ± 0.18 (56)	
$SiC + 50\%ZrB_2 + 10\%B_4C$	3.59 ± 0.12 (3)	3.51 ± 0.15 (3)	_ ` `	
Al ₂ O ₃ -998	$3.5\pm0.05(5)$	3.6 ± 0.06 (5)	$3.57 \pm 0.22 (135)$	
Y-PSZ	5.7 ± 0.17 (5)	5.9 ± 0.19 (5)	_	

[±] Standard deviation.

^a The number of specimen tested (in parentheses).

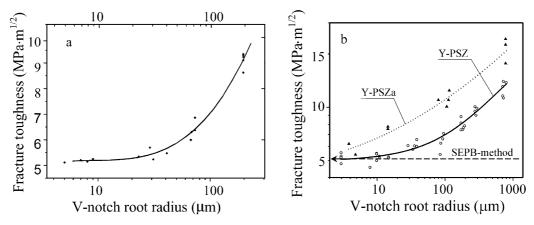


Fig. 3. Effect of V-notch root radii on the K_{Ic} values for Si_3N_4 (a) and Y-PSZ (b) ceramics.

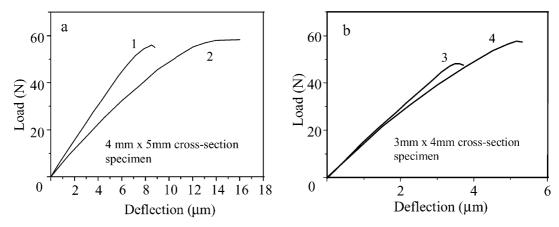


Fig. 4. Load–deflection diagrams for notched $Si_3N_4 + 30\%$ SiC + 3% MgO (a) and SiC + 50% $ZrB_2 + 10\%$ B_4C (b) specimens tested at room temperature (1, 3) and at 1400 °C (2, 4).

presented in Figs. 4 and 5. The results of micro-Raman analysis are given in Fig. 6. The comparative data on SEVNB and SENB results are summarized in Tables 4–6. High-temperature test results are cited in Table 7.

It is useful to start the analysis with emphasis on the K_{Ic} values obtained in three- and four-point flexure (Table 3). The data presented in this table demonstrate good agreement between our results and the average results of RRFT'97 [6], which confirms sufficient accu-

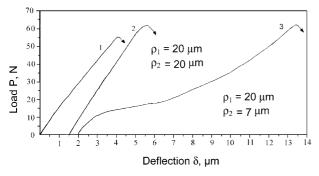


Fig. 5. Load–deflection diagrams for notched SiC+50%- $TiB_2+10\%B_4C$ specimens with V-notch root radiuses ρ_1 and ρ_2 are equal (1 and 2) and differed (3) in values on them opposite sides.

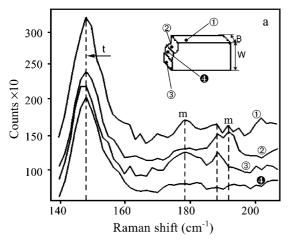
racy of our test procedures. The comparison of the data presented in Table 3 also points to the fact that essential differences between three- and four-point flexure results are absent. A similar conclusion was also made elsewhere [18] for ceramic matrix composites. Consequently, both test methods might be considered identical. Moreover, four-point flexure can be more easily applied in practice because it does not require a precise placement of specimens on the bearing rollers, which is difficult to achieve without a trained operator. On the other hand, three-point flexure tests can utilize small-size specimens, which is advantageous for materials science research.

The analysis of results (Fig. 3) shows that a decrease in the V-notch radius of a specimen leads to an essential decrease in the $K_{\rm Ic}$ values for ${\rm Si_3N_4}$ and Y-PSZ ceramics (similar relation was also observed for other materials [2]). It is interesting to note that the fracture toughness-notch root radius curve for ${\rm Si_3N_4}$ (Fig. 3a) flattens in the vicinity of a small notch root radius as compared to that of the Y-PSZ (Fig. 3b). Such an effect is probably determined by different sensitivity of these materials to stress concentrations because of differences in their

grain sizes (\sim 4 µm for Si₃N₄ and > 1 µm for Y-PSZ) as it was mentioned elsewhere [19]. It should be emphasized that only for notch root radii less than 5–7 µm [7] the $K_{\rm Ic}$ values for Y-PSZ ceramics agree with SEPB data (similar behavior is also typical of fine-grained alumina [20]. A similar conclusion follows from [6], where an attempt was made to relate the V-notch radius values required for the correct determination of $K_{\rm Ic}$ values to the averaged values of this parameter obtained in the RRFT'97 studies. As follows from Fig. 3a, the decrease in $K_{\rm Ic}$ values for Si₃N₄ ceramics occurs with a decrease in notch radii down to about 30 µm, which does not

contradict the results of sintered SiC whisker-reinforced Si₃N₄ [18] tests. A similar tendency is also typical of SiC ceramics [19].

The examination of the specimens fractured in the tests revealed a fracture crack that propagated from the points where "additional" stress concentrators were present. This observation also confirms the assumption that the fracture of a loaded ceramic specimen starts from a small crack ahead of a machined notch root [19]. In this context, it is interesting to mention that $K_{\rm Ic}$ magnitudes are influenced by the sharpness of a notch root rather than by its shape [7].



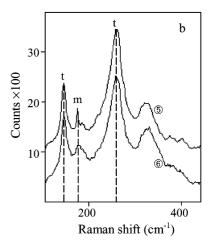


Fig. 6. Raman spectra of Y-PSZ specimens fractured by the SEVNB method (a) and by indentation (b): nonfractured specimen surface (1), diamond saw surface (2), fracture surface (3), razor blade surface (4), indentation edge (5), and indentation center (6).

Table 4 $K_{\rm Ic}$ values for ceramics obtained by SEVNB and SENB methods (MPa m^{1/2})

Material	Test method		Flexure (points)	Index φ	Brittleness measure, χ
	SEVNB	$SENB^a$			measure, _A
Y-PSZ	5.14±0.29	9.54±0.47	Four	0.538	1
Soda limit glass	0.66 ± 0.07	1.18 ± 0.09	Four	0.562	1
$SiC + 50\%ZrB_2 + 10\%B_4C$	3.52 ± 0.08	6.24 ± 0.37	Three	0.564	1
Si_3N_4	5.17 ± 0.06	9.12 ± 0.29	Three	0.567	1
SSiC	2.61 ± 0.18	4.42 ± 0.23	Four	0.590	1
$Si_3N_4 + 30\%SiC + 3\%MgO$	2.27 ± 0.14	2.49 ± 0.16	Three	0.920	0.88
Mg-PSZ (TS-grade)	9.44 ± 0.12	10.2 ± 0.27	Four	0.925	0.41
La _{8.8} Ca _{0.2} CoO ₃	2.2	2.2	Three	1	0.25

^a The width of notch is 0.2-0.3 mm.

Table 5 $K_{\rm Ic}$ values for single crystals obtained by SEVNB and SENB methods (MPa m^{1/2})

Single crystals	Peculiarity	Elastic modulus (GPa)	Test method	Test method		Index φ
			SENB	SEVNB	measure, χ	
Zirconia	Partially stabilized (3% Y ₂ O ₃)	245	9.33±0.95	10.33 ± 2.17	1	0.9
Alumina	Specimen axis 45° to optical axis of crystal	403	2.31 ± 0.34	2.45 ± 0.29	1	0.94
	Specimen axis 90° to optical axis of crystal	410	3.19 ± 0.53	2.85 ± 0.50	1	1.12

Table 6 $K_{\rm Ic}$ values for Si₃N₄/Si₃N₄ + n% TiN laminated composites ($\chi = 1$) obtained by SEVNB and SENB methods (MPa m^{1/2})

Content (n) of TiN layers ^a (%)	Test method	Average value of K_{IC} (MPa m ^{1/2})			
	SEVNB		SENB		
	Four-point flexure, 20/40 mm	Three-point flexure, 16 mm	Three-point flexure, 16 mm		
	$K_{\rm IC}$ (MPa m ^{1/2})	$K_{\rm IC}$ (MPa m ^{1/2})	$K_{\rm IC}$ (MPa m ^{1/2})		
10	6.61	6.80	6.27	6.56±0.19	
	6.45	6.37	5.74	6.19 ± 0.30	
	5.95	6.20	6.08	6.08 ± 0.08	
	5.26	5.59	7.03	5.96 ± 0.71	
	5.42	5.48	8.41	6.43 ± 1.32	
	5.26	5.90	4.94	5.37 ± 0.36	
	6.45	6.03	6.01	6.16 ± 0.19	
	5.87	5.71	5.85	5.81 ± 0.07	
Average	5.91 ± 0.46	6.01 ± 0.36	5.99 ± 0.41		
30	8.57	8.11	9.15	8.66 ± 0.58	
	9.84	9.31	9.28	9.48 ± 0.24	
	8.91	8.20	9.09	8.73 ± 0.36	
	9.63	7.87	8.07	8.52 ± 0.74	
Average	8.99 ± 0.75	8.37 ± 0.47	8.90 ± 0.41		

^a Average thickness of layers is 0, 185 mm.

The SEVNB method (see, e.g., [6]) is usually compared with the SEPB, SCF, and CNB methods. But it is also feasible to compare SEVNB and SENB data (Table 4), paying attention to the fact [21,22] that the evaluation of inelastic materials gives fracture toughness results which practically coincide with those for the specimens with sharp and blunt stress concentrators. Table 5 summarized the index of sensitivity to stress concentrations, φ , equal to the ratio of the $K_{\rm Ic}$ values obtained by the SEVNB and SENB methods, and the inelasticity of ceramics described by the brittleness measure χ [22]. The latter is equal to the ratio of the specific elastic energy accumulated in ceramics by the moment of fracture to the total energy spent for its deformation. Brittleness measure values were determined from stress-strain curves obtained in four-point flexure of solid (without stress concentrators) specimens. The analysis of these data confirms the existence of relationship between φ and χ . In the tests of elastic materials ($\chi = 1$), φ is about 0.6, and for inelastic materials (φ < 1), it exceeds 0.9. But this conclusion is correct only for monolith ceramics and ceramics particulate composites. For single crystals (Table 5) and ceramic laminated composites (Table 6) the picture is different and above-mentioned dependence is not observed.

Almost all the studies on the deformation behavior of V-notched ceramic specimens with a φ value of about 0.6, produced linear load-deflection diagrams or diagrams with small nonlinearity (e.g., curves 1 and 3 in Fig. 4), which is associated with a comparatively slow crack growth that is permissible in accordance with [23].

Table 7 High temperature fracture toughness test results (SEVNB method)

Materials	K _{IC} (MPa m ^{1/2})			
	20 °C	1300 °C	1400 °C	
Si ₃ N ₄	5.5±0.1	4.2±0.3	_	
$Si_3N_4 + 30\%SiC + 3\%MgO$	2.27 ± 0.1	_	2.68 ± 0.1	
$SiC + 50\%ZrB_2 + 10\%B_4C$	3.52 ± 0.1	3.63 ± 0.3	3.70 ± 0.1	
Si ₃ N ₄ [17] ^a	5.6 ± 0.5	5.0 ± 0.4	_	

^a The notches were produced by diamond saw with V-shaped tip.

But in some tests of ceramics particulate composites, the situation was different, especially if the sharpness of a V-notch was not uniform (V-notch root radii on the opposite sides of the specimen are not equal). Several load-deflection diagrams can have an unusual shape because fracture initiates in the vicinity of the notch root with a smaller radius, where higher stress concentrations are present (Fig. 5).

To complete the analysis of Table 4, we should note that in fracture toughness tests of TS-grade zirconia ceramics, the index φ was lower than unity. At the same time, the comparison of SENB and SEPB data for these ceramics (if the annealing of specimens was not performed at temperatures exceeding the boundary of monoclinic-tetragonal transformation) could give a ratio of $K_{\rm Ic}$ values more than unity. For example, this ratio was 1.24 [24], which was probably caused by the phase transformation in the area of the initial crack nucleation, when the specimen was prepared for SEPB. Therefore, it was interesting to investigate the phase

state on the fracture surfaces of zirconia specimens tested by the SEVNB method. The phase state was analyzed by micro-Raman spectroscopy (Fig. 6), which demonstrates, in contrast to fracture or saw notch surfaces (Fig. 6a) or the edge of indentation (Fig. 6b), a polished V-notch surface does not contain the monoclinic phase (there is no effect inducing the phase transformation). This is an important advantage of the SEVNB method in comparison with other fracture toughness test methods for zirconia and similar ceramics.

The comparison of high-temperature $K_{\rm Ic}$ evaluations for Si₃N₄ ceramics, presented both in Table 7 and in Ref. [18], reveals their similarity. However, in our case, in contrast to [18], fracture of the V-notch surface did not occur and we did not observe active oxidation in the vicinity of the notch. It is noteworthy that in SEPB tests of similar ceramics [18], oxidation induced the blunting (healing) of the initial sharp crack and, as a consequence, the fracture toughness results varied.

It is necessary to emphasize the increase in the fracture toughness of ceramic particulate composites with test temperatures (Table 7). The φ value varies moderately with this increase (0.81 for $Si_3N_4 + 30\%$ SiC + 3% MgO and $0.71 \text{ for SiC} + 50\% \text{ ZrB}_2 + 10\% \text{ B}_4\text{C}$). The changes in the load-deflection diagrams of these ceramics with test temperatures are not so pronounced (Fig. 4) and are connected with a certain increase in their inelastic deformation. It should be noted that the load-deflection diagrams for Si₃N₄ + 30%SiC + 3% MgO unnotched specimens and specimens with wide notches were linear. The oxidation layers on fractured specimens are not strong and cannot affect the fracture toughness of the ceramics studied in high-temperature SEVNB tests. And, probably, the blunting of a stress concentrator does not occur as in [18], where in SEPB tests crack healing and a considerable increase in K_{Ic} values, even at 1200 °C, were observed.

4. Conclusions

The test data confirm that the SEVNB method can be easier applied in practice and can be used for the majority of advanced ceramics and ceramic particulate composites at different temperatures and in the oxidation environment. The SEVNB data for ceramics and ceramic particulate composites are independent of the flexure type and exhibit small scatter. For laminated ceramic composites them are also independent of width of the stress concentrator. Therefore it commands the attention of engineers involved in both certification testing and materials science research. It was found that the ratio between SEVNB and SENB data equaled about 0.6 for elastic ceramics and ceramics particulate composites and over 0.9 for inelastic ones. The polishing-out of a V-notch root does not damage the surface layer formed on a zirconia; therefore, there are no residual stresses, phase transformations or other effects, which often accompany the formation of stress concentrators by other procedures.

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