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A SPECIFIC FEATURE IN THE FRACTURE OF POLYCRYSTALLINE ZIRCONIA CERAMIC

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The effect of indentor load F and V-notch tip radius r on the crack resistance factor K_{1c} of ZrO_2 + 3 mol.% Y_2O_3 ceramic is studied by microindentation and bending methods. Materials composed of 100% tetragonal phase (T-phase) and 70 – 80% T-phase were used. In both materials, an increase in F and a decrease in r cause a decrease in K_{1c}. The higher sensitivity of K_{1c} to F and r is observed in 100% T-phase specimens. This effect is explained by the involvement of martensite-type phase transformations.

In recent years, there has been increasing interest in the physicomechanical properties of ceramic materials. The reasons for this are, first, the ever-increasing production rate and range of application of engineering and instrumental ceramics and, second, the use of nanocrystalline powders in ceramic technology, which is essential for improvement of physicomechanical properties and, what is more important, the stability of ceramic materials. The latter circumstance has stimulated efforts in physical studies owing to which the mechanical properties of ceramic materials became controlled parameters, correspondingly with a wider range of their application [1]. Experimental data were obtained on these materials with a satisfactory reproducibility of measurements using various methods and laboratory techniques [2].

Our goal in this work was to study the physicomechanical properties of a ceramic based on ZrO_2 with 3 mol.% Y_2O_3. The ceramic was prepared by sintering nanocrystalline powders (Tosoh) with a crystallite size smaller than 30 nm and powders with a crystallite size smaller than 100 nm (available from the Vol'nogorskii Mining and Smelting Plant, VMSP). A technology based on cold isostatic pressing and special sintering regime was used to prepare test specimens with a standard size of 3 × 4 × 40 mm.

It was established by x-ray diffractometry that the Tosoh specimens were of tetragonal (T) phase whereas the VMSP specimens were of 70 – 80% T-phase and the rest were monoclinic (M) and cubic (C) phases.

Microindentation method and three- and four-point bending techniques using notched specimens were employed to study the crack resistance factor K_{1c} as a function of the indentor load F and the V-notch diameter d. Indentation tests were carried out using a standard pyramidal diamond-tipped Vickers indentor in the load range of $F = 40 – 300$ MPa. Based on the measured values of the impression diameter $2a$ and the radial crack length c, the crack resistance factor K_{1c} was determined using relation (1) [3]

$$K_{1c} = 0.018 \left(\frac{E}{H_V} \right)^{0.5} \frac{F}{c^{1.5}}, \tag{1}$$

where E is the elasticity modulus and H_V is the Vickers hardness.

In bending tests, the K_{1c} factor was measured by the SEVNB (single-edge-V-notch beam) method using a V-notch 0.8 – 1.6 mm deep b [4]. In the final stage, the V-notch was trimmed (usually a razor blade was used) to impart a curvature of radius d to the notch tip. The radius d was varied in the range of 3 – 200 μm. In the three-point bending technique the factor K_{1c} was determined by the formula [2]

$$K_{1c} = \frac{3F_{max} S_0}{2BW^{1.5}} d^{0.5} g(\alpha), \tag{2}$$

where W is the specimen thickness, F_{max} is the load at failure, and S_0 is the load-point distance,

$$\alpha = \frac{b}{W} = 0.45 – 0.55,$$

$$g(\alpha) = \frac{1.99 - \alpha(1-\alpha)(2.15 - 3.93\alpha + 2.7\alpha^2)}{(1-2\alpha)(1-\alpha)^{1.5}}.$$

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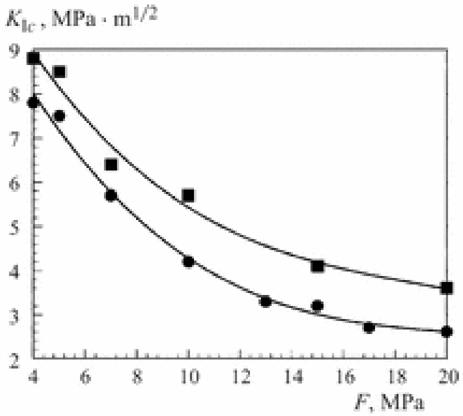

Fig. 1. The crack resistance factor K_{Ic} plotted as a function of the indentor load F: *1*) ceramic with 100% T-phase; *2*) ceramic with 70 – 80% T-phase.

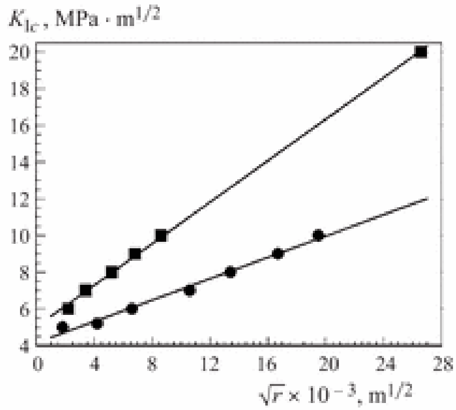

Fig. 2. The crack resistance factor K_{Ic} plotted as a function of the V-notch tip radius r: *1*) ceramic with 100% T-phase; *2*) ceramic with 70 – 80% T-phase.

In the four-point bending technique, the corresponding expression is

$$K_{Ic} = \frac{F_{max}}{BW^{0.5}} \frac{S_0 - S_1}{w}, \quad (3)$$

where S_1 is the load-point distance,

$$\alpha = \frac{b}{W} = 0.2 - 0.3,$$

$$g(\alpha) = 1.9887 - 1.326\alpha - \frac{(3.49 - 0.68\alpha + 1.35\alpha^2)(1-\alpha)\alpha}{(1+\alpha^2)}.$$

Shown in Fig. 1 is the crack resistance factor K_{Ic} plotted as a function of the indentor load. As is seen, for both types of ceramic, the indentor load increment of about 300 MPa causes a decrease in K_{Ic} by a factor of 2.5 to 3. The observed behavior in these ceramics differs from that of high-strength polycrystals, single crystals, and glasses [5, 6] for which K_{Ic} is constant over a wide range of F.

The factor K_{Ic} plotted as a function of the V-notch tip radius using bending test data is shown in Fig. 2 (note that the three-point and four-point tests gave identical results). The experimental points in Fig. 2 are well fitted by straight lines plotted on the coordinates K_{Ic} versus $\sqrt{r}$. One will note the higher sensitivity of the crack resistance factor of the single-phase ceramic to the notch tip radius. As is known, two criteria — a force criterion and an energy criterion — are considered in the theory of crack nucleation and growth [7]. The force criterion is based on the following condition: the local stress at the crack mouth σ_i is assumed to be higher than the theoretical stress. The energy criterion is based on the use of the surface energy γ.

In fracture mechanics, the stress intensity factor K_{Ic} is used as a criterion of local fracture. The maximum stress at the crack tip is determined as

$$\sigma_{max} \approx \sigma \sqrt{\frac{c}{r}} \approx \frac{K}{\sqrt{r}}, \quad (4)$$

where c in the crack length, σ is the stress, and r is the crack tip radius.

The condition for crack propagation is

$$K \geq K_{cr} \cong \sigma_0 \sqrt{r}, \quad (5)$$

where $\sigma_0 = 0.1E$ is the theoretical stress.

Using the V-notch to as a model for the crack profile makes it possible to establish a relation between the tip radius (for a constant notch depth) and the fracture toughness K_{Ic}.

It follows from the above relationships that the bending test method allows one to obtain a relationship $K_{Ic} = f(r)$ in qualitative agreement with the theory. This implies that relaxation is a process of minor (if any) importance for the stress behavior at both the edges (no plastic strain!) and the mouth of the crack. The different crack resistance in different batches may be due to the difference in phase composition. As is known, mechanical stress initiates transformation toughening. In the crystalline partially stabilized zirconia (PSZ) this process involves a transition from the tetragonal to the monoclinic phase and it is the most intense at the crack mouth where the stress concentration is maximum. Within this model, the highest crack sensitivity is expected in specimens with a higher percentage of the tetragonal phase, in agreement with experiment.

A feature in the relationships $K_{Ic} = f(r)$ in Fig. 2 deserves particular note. The two plots when extrapolated as $r \to 0$ give roughly the same intercept value of K_{Ic} at

4.7 ± 0.5 MPa $\cdot$ $m^{1/2}$. This fact may be explained by the model used: for values of r smaller than the crystallite diameter, the role of the tetragonal phase as a toughening factor is minimum.

The qualitative agreement of our results with the theoretical model (5) of fracture mechanics provides evidence that the nucleation of new cracks and the propagation of cracks preexisting on the ceramic surface is not an easy process. This conclusion follows from the fact that in ceramic materials the crack tip radius is roughly the same as the α-interatomic spacing. In the absence of plastic strain this parameter should remain constant. Consequently, one should not expect a relationship to exist between the fracture toughness factor and the V-notch tip radius since $r \gg a$ in all cases. The inhibition of crack nucleation and propagation in PSZ ceramics can be effected only via transformation toughening. The mechanical stress at the crack tip initiates the tetragonal-to-monoclinic phase transition; of the two phases, the latter has a larger volume, which imposes a constraint on the crack growth. For the same reason the crack nucleation is made difficult on a bent specimen's surface, as was shown experimentally in [8].

Another important finding in our study was that the fracture toughness factor K_{1c} was found to be dependent on the indentor load. The crack length c as a function of the indentor load F is shown in Fig. 3; plotted on the coordinates $\sqrt{c}$ versus F, it is approximated by straight lines.

A similar dependence of the crack length on the applied load is inferred from a model of steady crack growth [7]:

$$c = \frac{P^2}{2\pi E\gamma}, \tag{6}$$

where P is the tensile point load applied to both crack edges.

On reaching a critical load value, the crack growth is initiated. The crack is growing uniformly, with its length being proportional to the square of the load, which is borne out experimentally (see Fig. 3).

The dependence of the fracture toughness factor K_{1c} (which is a parameter of the material) on the indentor load calls for a caveat in using the microindentation method for studying the toughness properties of PSZ ceramics.

The observed abnormality of crack propagation has also a geometrical aspect. As is known, the "radial" PSZ cracks are distributed over the indentation depth. This specific feature of the crack formation in zirconia, uncommon to other materials, is explained by a mechanism involving the activation of the T – M phase transition not only in the mouth of the crack, but also in a zone located directly beneath the

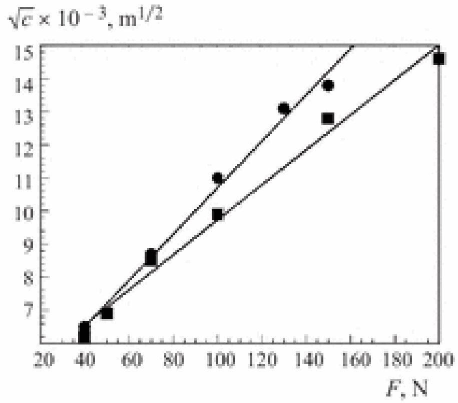

Fig. 3. The crack length plotted as a function of the indentor load F: *1*) ceramic with 100% T-phase; *2*) ceramic with 70 – 80% T-phase.

indentor as it penetrates into the material. The quasi-hydrostatic pressure produced during the indentation amounts to about 10 GPa, which is higher than the pressure for polymorphic transition from the monoclinic to the rhombic phase (4 GPa). The initial state to test specimens was the tetragonal phase; one would assume therefore that a T – C phase transition might occur in a zone affected by the indentation pressure accompanied by a decrease in volume.

It should be noted in conclusion that the microindentation method can be used to study the strength behavior of PSZ ceramics, albeit with the caveat that F = const in carrying out experiments. Still, the data obtained under these conditions should be regarded as tentative. More experimental work is needed to substantiate the use of this method for further studies.

REFERENCES

1. A. I. Gusev, *Usp. Fiz. Nauk*, **168**(1), 55 (1998).
2. G. A. Gogotsi, *Ogneup. Tekh. Keram.*, No. 1, 7 (1998).
3. T. Miyoshi, et al., *Trans. Jap. Soc. Mech. Eng.*, **51A**, 2489 (1989).
4. T. Nishida, Y. Hanaki, and G. Pezzotti, *J. Am. Ceram. Soc.*, **77**(2), 606 (1994).
5. G. R. Anstis, P. Chanticul, B. R. Lawn, and D. B. Marshall, *J. Am. Ceram. Soc.*, **64**(9), 333 (1981).
6. L. I. Pyatykhin, A. G. Val'ko, and I. I. Papirov, *Fracture Toughness As Determined by the Indentation Method, Review Paper* [in Russian], TsNIIatominform, Moscow (1987).
7. V. I. Vladimirov, *The Physical Nature of Metal Fracture* [in Russian], Metallurgiya, Moscow (1984).
8. D. B. Marshall and M. R. James, *J. Am. Ceram. Soc.*, **63**(3), 215 (1986).