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Determination of the ATZ ceramics with different SiO_2 contents tensile strength by the „brazilian test“ method

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The possibility of using the „Brazilian test“ method to determination the tensile strength σ_t of composite zirconia ceramics small-size samples were demonstrated and the reliability of the obtained σ_t values were confirmed. It was found that the dependence of the tensile strength of alumina toughened zirconia (stabilized by calcium oxide) with silica addition ($CaO-ATZ+SiO_2$ ceramics) on SiO_2 concentration in them has a maximum ($\sigma_t = 450$ MPa, at $C_{SiO_2} = 5$ mol.%). The observed toughened is explained by an increase in the transformability of the tetragonal t-ZrO_2 phase and, accordingly, by an enhanced role of transformation toughening when SiO_2 is adding into the ATZ ceramics.

Keywords: ATZ ceramic, tensile strength, „Brazilian test“, phase transformations, transformation toughening.

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1. Introduction

Zirconium ceramics stabilized in the tetragonal phase has a high fracture viscosity, conditioned by a mechanically induced tetragonal-monoclinal $t \to m$ phase transition (the transformational strengthening mechanism [1,2]). Stabilization of the tetragonal phase of zirconium dioxide t-ZrO_2 at room temperature is usually performed by injecting the Y_2O_3 additive. The use of alternative stabilizers (CeO_2 or CaO) somewhat deteriorates the mechanical properties, but significantly increases the resistance of zirconium ceramics to low-temperature break degradation [3,4]. Injection of finely-divided particles of harder materials into zirconium ceramics enables the dispersion strengthening mechanism [5], related to dissipation of crack energy when it goes around an obstacle. The most widespread at present are zirconium ceramics reinforced with aluminum oxide (ATZ-ceramics) [6]. Thanks to the unique mechanical properties combined with a high thermal, chemical and radiation resistance, as well as biological inertness, the zirconium and ATZ-ceramics of engineering purpose have a very broad range of practical applications from machine building to medicine [7].

Regardless of application field of zirconium ceramics (stabilized in the tetragonal phase), their mechanical properties are of considerable importance. Variation of composition (stabilizer type, correlation of concentration of ATZ-ceramics components) and the microstructure design (size of crystallites t-ZrO_2 and reinforcing elements) allows for controlling the correlation of hardness values H and fracture viscosity K_C (on $H = 18$ GPa and $K_C = 2.7\,\mathrm{MPa}\cdot\mathrm{m}^{1/2}$ [8] up to $H = 10.4$ GPa and $K_C = 16\,\mathrm{MPa}\cdot\mathrm{m}^{1/2}$ [9]). Injection of additional components (e.g., SiO_2 or $SrAl_{12}O_{19}$) makes it possible to attain not only an improved correlation of the values of H and K_C [10], but also a manifestation of plasticity features of zirconium dioxide-based composite ceramics at room temperatures [11–14].

Brittle fracture under deformation not only restricts the machining methods and application field of articles made of zirconium ceramics and composites on its basis, but also significantly complicates the diagnostics of strength characteristics. This particularly applies to determination of ultimate tensile strength σ_t. Measurement of σ_t of brittle materials is associated with technical difficulties in sample preparation and sample alignment strictly along the deformation axis [15]. Along with that, the value of σ_t for brittle materials can be measured indirectly using the „Brazilian test“, which has been successfully used to study rocks and concretes [16,17].

The paper is aimed at verification of applicability of the „Brazilian test“ for estimation of tensile strength of small-size samples of ATZ-ceramics and study of influence of the SiO_2 additive on its strength properties.

2. Experimental procedure

The samples of zirconium ceramics (stabilized with calcium oxide), reinforced with aluminum oxide having a different content of silicon dioxide ($CaO-ATZ+SiO_2$) were made using ZrO_2 (Sigma-Aldrich), Al_2O_3 (Hongwu), CaO (Reachem) and SiO_2 powders (Sigma-Aldrich). The correlation of the initial (before grinding) molar concentrations of $ZrO_2:CaO:Al_2O_3$ was maintained constant — $88:6.2:5.8$ in compliance with [10]. Molar concentration of

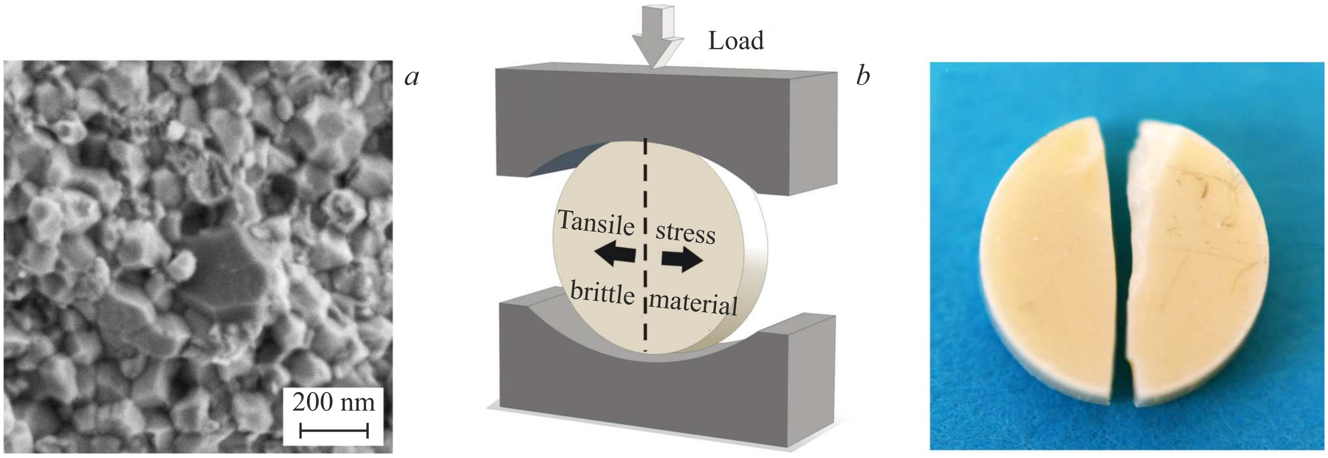

Figure 1. Typical SEM-image of the chip surface of $CaO-ATZ + SiO_2$-ceramics — *a*, schematic image of the „Brazilian test“of brittle samples — *b* and photograph of the $CaO-ATZ + SiO_2$-ceramics sample after a „Brazilian test“of the ultimate tensile strength — *c*.

SiO_2 in relation to the obtained mixture was varied from 0 to 6 mol.%. Dispersion of the prepared powder mixtures in distilled water, grinding in the Pulverisette planetary mill (Fritsch), mixture drying, sample forming in the shape of cylinders with the diameter of 10 and thickness of 2 mm, as well as their sintering were performed in full compliance with the conditions outlined in [10].

Relative density ρ of the samples was monitored by the Archimedes method using a laboratory balance (sensitivity limit 0.001 g). Phase composition was monitored by analyzing the diffraction patterns recorded by means of the D2 Phaser X-ray diffractometer (Bruker AXS). The microstructure was visualized for analyzing the sizes of ZrO_2 and Al_2O_3 crystallite using a Merlin high-resolution scanning election microscope (Carl Zeiss). All the made samples had a relative density ρ not lower than 95%. The relative fraction of the tetragonal phase of zirconium dioxide was not less than 93%. Sizes of ZrO_2 and Al_2O_3 crystallites did not exceed 100 and 250 nm respectively. As an example, Fig. 1, *a* shows a SEM-image of the chip of a sample (Al_2O_3 crystallites have a darker hue). The chip surface has traces of trans-granular breakdown of ZrO_2 crystallites. This indicates a high degree of intergranular interaction.

The „Brazilian test“was used for indirect determination of ultimate tensile strength σ_t. According to the numerous publications, compression of a short cylinder (placed on the side surface) causes tension stresses directed normally to the diametral plane coaxial to compression (Fig. 1, *b*). When the critical values of tension stresses are reached, a crack forms in the diametral plane of the samples of brittle materials under testing and the sample usually splits in half (Fig. 1, *c*). Correlation of the ultimate load and geometrical parameters of the sample makes it possible to estimate the quantity σ_t. The ASTM D3967−95a standard prescribes determination of the ultimate tensile strength using the formula

$$\sigma_t = \frac{2F}{\pi D t}, \quad (1)$$

where F is the ultimate load at which the brittle sample has broken down, D is sample diameter, t is its thickness.

It should be noted that, according to the ASTM D3967−95a standard, the diameter of the tested disk (rock sample) must be not less than 50 mm, while the disk thickness/diameter ratio must be within the range of 0.2−0.75. However, the technology for the making of new materials is usually mastered on smaller samples. The authors of [18] have shown that the „Brazilian test“ and correlation (1) can be used to estimate the values of σ_t for not only concrete and rocks but also for other brittle materials, and smaller samples can be used for these purposes (diameter of 10−15 and thickness of 1−10 mm). In compliance with this, the loading diagrams for the studied (cylindrical) samples were presented in $\sigma(\varepsilon)$ coordinates. For this, stress σ was calculated using the expression (1), assuming F as the current load, while deformation ε was calculated using the formula

$$\varepsilon = \frac{\Delta D}{D} \times 100\%, \quad (2)$$

where ΔD is the current change of the diameter in the direction coaxial to compression of the tested disk.

Deformation by way of the „Brazilian test“ was performed using a floor-standing MTS 870 Landmark double-column servohydraulic testing system (MTS, USA). The rate of deformation (movement of the upper punch) in all tests was maintained constant — 1 μm/s.

3. Results and discussion

Fig. 2 brings into compliance the typical $\sigma-\varepsilon$-diagrams recorded during deformation of the samples of CaO-ATZ + SiO_2-ceramics (having a different silicon dioxide content) by the „Brazilian test“method.

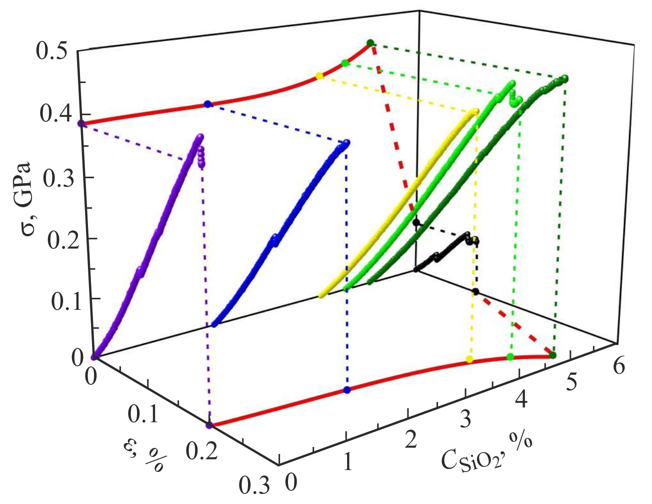

Figure 2. Diagrams of deformation of samples of $CaO-ATZ+SiO_2$-ceramics with a different SiO_2 content by the „Brazilian test“ method.

A projection of the limit values of σ and ε onto the corresponding coordinate planes allows for visualization of dependences of the ultimate tensile strength σ_t and ultimate deformation ε_c on concentration of silicon dioxide C_{SiO_2} in the ceramics under study. It can be seen that an increase of the silicon dioxide concentration from 0 to 5 mol.% is accompanied with a gradual increase both of the ultimate tensile strength and ultimate deformations. The following takes place in the range of $5\,\text{mol.\%} < C_{SiO_2} \leq 6\,\text{mol.\%}$: embrittlement of $CaO-ATZ+SiO_2$-ceramics and a sharp drop of the values of σ_t and ε_c (Fig. 2). The observed embrittlement of $CaO-ATZ+SiO_2$ ceramics at $C_{SiO_2} > 5\,\text{mol.\%}$ is fully consistent with the obtained data during deformation of similar ceramics by uniaxial compression and three-point bending [14], as well as during diagnostics of fracture viscosity [10].

The ultimate strength to ultimate deformation ratio for brittle materials (including zirconium ceramics) is equivalent to the Young modulus. Zirconium ceramics and their composites (having a small content of reinforcing additives) have the Young modulus $E = 200 \pm 15$ GPa [2,11,13,15,19]. Along with that, control of the phase composition (mechanically induced transformability of the tetragonal phase of zirconium dioxide) allows for increasing the ultimate strength [19]. Given the weak sensitivity of the Young modulus to type and concentration of t-ZrO_2 stabilizer [15,19,20], this is accompanied with a virtually proportional increase of ultimate deformation. As an example, Fig. 3 (the data marked with signs *1, 2* and *3*) shows the harmonized σ_t and ε_c correlations obtained in [15,19,20] by the conventional methods of tensile deformation of zirconium ceramics, stabilized with Y_2O_3 (in different concentrations) and CeO_2 oxides. It can be seen that the combination of these correlations is described by a linear function with a coefficient (slope ratio of the straight line), which corresponds to the Young modulus value of $E \approx 200$ GPa, typical for zirconium ceramics stabilized with yttrium oxide [15,19]. The same figure shows the correlations σ_t and ε_c obtained by the „Brazilian test“ method on the samples of $CaO-ATZ+SiO_2$-ceramics containing SiO_2 in the concentrations of 0, 2, 4, 4.5 and 5 mol.% (the data marked with signs *4*). It can be seen that one of the obtained correlations σ_t and ε_c for CaO–ATZ-ceramics ($C_{SiO_2} = 0$ mol.%), „falls“ on the drawn straight line. Accordingly, the values of σ_t and ε_c, obtained by the „Brazilian test“method, can be considered reliable, and the („Brazilian test“) method — suitable for diagnostics of the ultimate tensile strength of zirconium ceramics (sample diameter does not exceed 10 mm).

Injection of silicon dioxide into CaO–ATZ-ceramics causes a deviation of the σ_t and ε_c correlations from the straight line drawn in Fig. 3 (the data is marked with signs *4*). This means a gradual decrease of the Young modulus of $CaO-ATZ+SiO_2$-ceramics as its silicon dioxide concentration increases. The made conclusion is consistent with the data obtained by the nanoindentation method [13].

Proceeding to a discussion of the obtained results, we would like to note that tension stresses, originating in the diametral plane of a cylindrical sample under deformation by the „Brazilian test“ method, initiate tetragonal-monoclinal $t \rightarrow m$ phase transitions in the zirconium ceramics. This process is accompanied with a 4% volume increase and a 16% shear deformation, which partially compensates the tension stresses and prevents the crack origination and development. According to the data of [10 and 13], injection of SiO_2 into CaO–ATZ-ceramics facilitates an increase of transformability of the tetragonal phase of zirconium dioxide, i.e. it enhances the role of the transformational

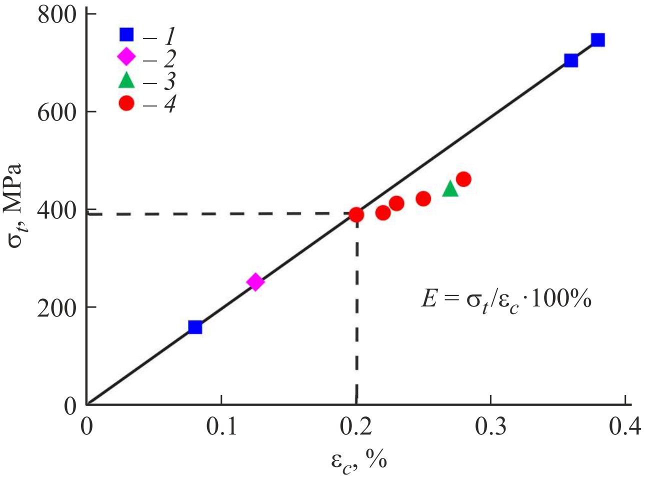

Figure 3. Correlations of ultimate tensile strengths σ_t and the corresponding ultimate deformations ε_c obtained on the $Y_2O_3-ZrO_2$ samples having a different content of Y_2O_3 — *1* and *2* (adapted from [19] and [15] respectively), CeO_2-ZrO_2 — *3* (adapted from [20]) and $CaO-ATZ+SiO_2$ with a different content of SiO_2 — *4*.

strengthening mechanism. This, in our opinion, is the reason for the revealed increase of the ultimate tensile strength and ultimate deformation of CaO$-$ATZ-ceramics upon injection of 4$-$5 mol.% of silicon dioxide (Fig. 2). However, exceeding of the threshold value of SiO_2 concentration in CaO$-$ATZ + SiO_2-ceramics ($C_{SiO_2} > 5$ mol.%) causes destabilization of the tetragonal phase of zirconium dioxide and abrupt embrittlement of ceramics.

4. Conclusion

We have demonstrated the applicability of the „Brazilian test" method for testing small-size samples of zirconium ceramics for indirect estimation of the ultimate tensile strength. Dependences of ultimate yield strengths σ_t and the corresponding ultimate deformations ε_c on concentration of silicon dioxide C_{SiO_2} have been established using the samples of CaO$-$ATZ + SiO_2-ceramics having same-type structures (and a different content of SiO_2) . It is shown that the maximum σ_t and ε_c correlation is achieved at $C_{SiO_2} = 5$ mol.%. According to the previous data, CaO$-$ATZ + SiO_2-ceramics with 5% content of silicon dioxide has an increased compression strength ($\sigma_c = 2.4$ GPa [13]) and a high correlation of hardness and fracture viscosity ($H = 10.9$ GPa, $K_C = 12.43\,\text{MPa}\cdot\text{m}^{1/2}$ [10]). The detected increase of ATZ-ceramics strength upon injection of SiO_2 additive is explained in terms of an increase of mechanically induced transformability of the tetragonal phase of zirconium dioxide and, respectively, enhancement of the role of the transformational strengthening mechanism.

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Conflict of interest

The authors declare that they have no conflict of interest.

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