Stress-strain behavior of high porous zirconia ceramic

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Annotation

In this work, we studied the deformation behavior of ZrO_2 ceramics stabilized with 5.5 wt.% Y_2O_3 with a porosity of 4 - 42% during diametral compression tests ("Brazilian" test). It has been shown that with an increase in porosity from 4 to 42%, the ultimate tensile strength in diametral compression decreases from 115 to 9 MPa. The ultimate strain before fracture decreases from 1 to 0.8%. The effective modulus also decreases with increasing of porosity. The analysis of the "stress-strain" curves showed that the deformation behavior of ceramics is influenced by both the volume and the morphology of the pore space. It been shown that two strain exponent were observed, which indicate a change of deformation mechanism of the ceramic during loading. X-ray diffraction analysis carried out from the front surface of fracture fragments of samples with porosities of 4, 17, and 42% showed that in these materials microstructural parameters such as the size of the coherently diffracting domains of the tetragonal phase and lattice microdistortion changes in comparison with the initial state after sintering. These materials microstructural parameters are changes non-uniformly, which indicate the inhomogeneity of the deformation of this brittle material during compression.

Keywords: ceramics, lattice microdistortion, porous, stress, zirconia

Kulcsszavak: kerámia, rácsos mikrotorzítás, porózus, feszültség, cirkónia

1. Introduction

The study of the deformation and fracture behavior in a brittle porous material under its mechanical loading has recently attracted considerable attention of researchers [1-5]. These studies are important for estimation of the fundamental base for the synthesis of new composite materials which can be used for extremely loaded conditions, as biomaterials, filters, and soundproof materials for various anti-noise devices. Mechanical strength is a key parameter for many ceramic applications where porous ceramic parts are subjected to compression, bending, tension and shear [6]. Also, these data can be very useful for the model study of mechanical properties and processes of fracture of rocks [7]. Today, most mechanical tests have been carried out on dense materials with a uniform structure. The processes of deformation and fracture of porous ceramic materials are being actively investigated, and in literature there are some results of measuring of elastic constants with different porosity [8]. The effects of nonlinear elasticity under mechanical loading of porous ceramics based on zirconia are described in [9]. It has been determined that deformation of materials with a complex internal structure, local strains due to relative displacements and deformations of its structural components play a significant role, which noticeably changes the elastic characteristics of materials. In the literature, this behavior is discussed in terms of the

transition from brittle fracture to quasi-plastic [10-13]. This was previously observed in the case of concrete [14], ferroelectric ceramics [15], and a glass or ceramic matrix composites [16]. Recently, a similar deformation behavior was observed for uniaxial compression tests of porous ceramics [17-18], as well as in bending tests of refractory materials [19]. At the same time, works in which other schemes of loading brittle materials are used are not enough. So the performance of direct tests for uniaxial tension of brittle materials is associated with the technical problem of applying tensile axial forces to the sample [20]. To determine the tensile strength of brittle materials, an indirect method for determining the tensile strength of the material, the so-called "Brazilian test", is successfully used, when tensile stresses are formed in the center of the sample [21-23]. The particular qualities of the stress-strain state of the samples during the Brazilian test continue to attract the interest of many researchers from the moment of its proposal. Investigations are carried out by various experimental [22-24] and numerical methods [25-28]. Nevertheless, investigations of the deformation behavior of ceramic materials based on

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zirconia in a wide range of changes in pore sizes and pore volume have been insufficiently conducted.

The aim of this work was to study the stress-strain behavior of porous zirconia ceramics with a wide range of porosity during diametral compression tests.

2. Materials and methods

To obtain experimental samples, we used zirconia powder (ZrO_2) stabilized with 5.5 wt% yttrium oxide (Y_2O_3). The study of the ZrO_2 -5.5 wt% Y_2O_3 powder (ZrO_2 (Y)) was carried out on a VEGA Tescan 3 SBH scanning electron microscope. The particle size distribution of the powder, including agglomerates, and a typical SEM image of the powder are shown in Fig. 1.

The powder is a finely dispersed mixture of agglomerated particles of irregular shape. The particle size of the powder varies from 0.1 to 2 μm , and the fraction of particles up to 0.5 μm is about 70%. The specific surface area of the powder (S_{sp}), measured on a SORBI-4.1 device, by low-temperature nitrogen adsorption by the 4-point BET method [29] is $8.05 \pm 0.085 \text{ m}^2/\text{g}$. According to the results of X-ray phase analysis, the powder consists of tetragonal and monoclinic phases of zirconia. The content of the monoclinic phase in the powder is 44%.

Experimental samples traditional methods of powder metallurgy were prepared. The plasticized (5% carboxymethyl cellulose (CMC) aqueous solution) powder was cold isostatic compaction with a hydraulic press under a pressure of 50 MPa and followed low-temperature annealing of the compacts to remove the binder was carried out at a temperature of 1100 °C with a heating rate of 1.5 °C/min in the MgO powder fill. The final sintering of the samples was carried out in a high-temperature air muffle furnace LHT "Nabertherm" according to the modes presented in Table 1. To obtain samples with different porosities, the sintering temperature and isothermal holding time were varied.

The pore structure of the sintered ceramics was studied on polished surfaces using VEGA Tescan 3 SBH scanning electron microscope. The determination of the average pore size and their size distribution was carried out using the ImageJ program; in this case, at least three images of the structure and at least 1000 measurements were used. The ratio of pores size smaller than 30 μm and over 30 μm of all investigated ceramics was calculated by the methods of stereometric metallography [30]. The two-dimensional distribution of pores by size was transformed into a three-dimensional distribution using the basic stereometric equation of Saltykov [30]. Zirconia phase fractions were quantified on sintered and fractured specimens using the X-ray diffraction analysis (XRD). XRD spectra were collected over a 2θ range between 20° and 80° using a powder diffractometer equipped with a Cu X-ray source with a step size of 0.05° with statistical accuracy better 3%. XRD line profile analysis was used to determine of the size of the coherently diffracting domain (D) and crystal lattice microdistortion ($\langle \epsilon^2 \rangle^{1/2}$) of tetragonal phase of zirconia. The size of the coherently diffracting domains (sCDDs) was calculated by the Scherrer equation [31] for the lines (111) and crystal lattice microdistortion was calculated according to the Stokes – Wilson equation [32] for the lines (004).

Fig. 1 Particle size distribution of zirconia powder. The insets show the SEM image of the powders

1. ábra A cirkóniumpor részecskeméret-eloszlása. A porok SEM felvétele

The porosity and phase composition of the specimens before diametral compression tests (in the initial state), depending on the sintering conditions, are given in Table 1.

Sample number	Sintering conditions, $T, ^\circ\text{C}; \tau, \text{h}$	Porosity, %	Phase composition, %
1	$T = 1600^\circ\text{C}, \tau = 1 \text{ h}$	4	$\text{ZrO}_2(t^*) - 80;$ $\text{ZrO}_2(c^{**}) - 20$
2	$T = 1500^\circ\text{C}, \tau = 1 \text{ h}$	17	$\text{ZrO}_2(t) - 80;$ $\text{ZrO}_2(c) - 20$
3	$T = 1400^\circ\text{C}, \tau = 3 \text{ h}$	29	$\text{ZrO}_2(t) - 88;$ $\text{ZrO}_2(c) - 9$ $\text{ZrO}_2(m^{***}) - \text{trace (less 3)}$
4	$T_{\text{cnek}} = 1400^\circ\text{C}, \tau = 2 \text{ h}$	33	$\text{ZrO}_2(t) - 93;$ $\text{ZrO}_2(c) - 6$ $\text{ZrO}_2(m) - \text{trace (less 3)}$
5	$T_{\text{cnek}} = 1350^\circ\text{C}, \tau = 1 \text{ h}$	42	$\text{ZrO}_2(t) - 95;$ $\text{ZrO}_2(c) - 4$ $\text{ZrO}_2(m) - \text{trace (less 3)}$

t^* – tetragonal phase ZrO_2 ;

c^{**} – cubic phase ZrO_2 ;

m^{***} – monoclinic phase ZrO_2

Table 1 Porosity and phase composition of ZrO_2 (Y) ceramic samples depending on sintering conditions

1. táblázat ZrO_2 (Y) kerámiatínták porozitása és fázisösszetétele a szinterelési körülményektől függően

The phase composition of ceramic samples sintered at temperatures of 1500 and 1600 °C is represented by high-temperature tetragonal and cubic phases of zirconia in a ratio of 80:20, respectively. With a decrease of the sintering temperature, the ratio of the tetragonal and cubic phases changes, with a decrease in the content of the cubic phase and the appearance of traces of the monoclinic ZrO_2 phase, Table 1.

Diametral compression tests (the Brazilian test) of ceramic samples with a diameter of $27.5 \pm 0.3 \text{ mm}$ and a height of $11.3 \pm 0.2 \text{ mm}$ were carried out on a universal testing machine "Instron" at a loading rate of 0.1 mm/min with automatic recording of the loading diagram "load - displacement" taking

into account the rigidity of the loading system. The calculation of stresses was performed according to [33].

3. Results and discussion

The pore structure of ZrO_2 (Y) ceramic samples with different porosity and pore space morphology is shown in Fig. 2. Interparticle isolated porosity of ceramics with a porosity of 4% (sample 1, table 1), is observed. The average pores size is $\approx 4 \mu m$ and the maximum pores size does not exceed $30 \mu m$, Fig. 2 (a). Two types of pores and a bimodal pore size distribution in the structure of ceramic samples with a porosity of 17% and higher (samples 2 - 5, table 1), are observed, Fig. 2 (b). In addition to interparticle porosity (1 - $30 \mu m$), the ceramic contains large interagglomerate pores of irregular shape with sizes of $30 - 80 \mu m$, the number of which increases with increasing porosity.

Fig. 2 Typical image of the porous structure: (a) - ceramic with porosity 4%; (b) - ceramic with porosity 42%. The insets show the pore size distribution of ceramics with different pore space morphology

2. ábra A porózus szerkezet tipikus képe: (a) - 4%-os porozitású kerámia; (b) - 42%-os porozitású kerámia. A különböző pórustér morfológiájú kerámiák pórusméret-eloszlása

The changes in the average pore size of the studied ceramics are shown in Table 2. It can be seen that with the porosity of the samples increasing, the average pore size ($\langle d \rangle$) increases from 4 to $6.4 \mu m$. In this case, with an increase in the average pore size, the size dispersion of powders also grows. Also, with an increase in porosity, an increase of the average size of both small $\langle d_1 \rangle$ and large pores $\langle d_2 \rangle$ is observed. From the histogram shown in Fig. 2 (a), it can be seen that in the sample with a porosity of 4%, pores larger than $30 \mu m$ are absent. In samples with a porosity of 17% and higher, the pore volume with sizes of pores larger than $30 \mu m$ increases with increasing porosity but does not exceed 10 vol.%.

Sample number	Porosity, %	$\langle d \rangle \pm 0.2 \mu m$	Sd	$\langle d_1 \rangle \pm 0.2 \mu m$	$\langle d_2 \rangle \pm 1 \mu m$	V_{por} (Size of pores <30 μm), %	V_{por} (Size of pores >30 μm), %
1	4	4.07	3.2	-	-	100	-
2	17	4.95	4.5	3.8	28.3	97	3
3	29	5.3	4.7	4.74	32.4	95	5
4	33	5.33	5.6	4.9	39	90	10
5	42	6.4	5.8	6.1	38.1	93	7

Table 2 Change in the average pore size in ceramics with different porosities
2. táblázat A kerámia próbatetek átlagos pórusméretének változása a porozitástól függően

Fig. 3 (a) shows the “stress-strain” curves during diametral compression tests of ceramic specimens with different porosities. The diametral compression experiments showed that the behavior of all specimens upon loading was typical as for brittle materials. An analysis of the deformation curves showed that for samples with porosities of 4 and 17% were deformed elastically up to its fracture. The deformation curves of specimens with a higher porosity are showing a deviation from linearity before their fracture. The deviation from linearity of specimens with a porosity of 27% and higher was observed in other works [8, 12] for other loading schemes of porous ceramics and may be associated with the appearance and accumulation of defects in the form of microcracks during loading, which is representative of the quasi-brittle fracture of porous ceramic materials.

Re-plotting of the « $\sigma - \epsilon$ » dependences in double logarithmic coordinates $\ln(\sigma/\sigma_0) - \ln(\epsilon)$, they were transformed into two parts, which can be approximated by linear functions with varying inclination to the X-axis ($\ln(\epsilon)$), and therefore, with different exponent of strain hardening n of the Hollomon equation [34]. The inset in Fig. 3 (a) shows a representative curve with different exponents of strain hardening n_1 and n_2 . The exponent of strain hardening n_1 , calculated from linear functions, increase from 1.45 to 1.7 with an increase of samples porosity from 4 to 33% and in a sample with a porosity of 42%, a decrease in n_1 is observed, Fig. 3 (b). The exponent of strain hardening n_2 practically does not change with an increase of the porosity of the samples and is equal to ~ 1.2 .

It should be noted that on all dependences « $\ln(\sigma) - \ln(\epsilon)$ » the strain exponents n are large 1, which probably corresponds to the nonlinear elastic behavior of ceramics under load with a change of porosity. In this case, the change in the angle of inclination of the linear sections on the curves « $\ln(\sigma) - \ln(\epsilon)$ »

may indicate a change in the mechanism of deformation of the ceramic during loading. The change in the slope on the curves for ceramics with porosities of 4 and 17% can be associated with microcracking caused by the tetragonal-monoclinic phase transformation during loading. On the fracture surface of samples with porosity of 4 and 17% according to the X-ray phase analysis the formation of a monoclinic phase with a volume content of 30% and 12%, respectively, are observed.

Fig. 3 Deformation curves of $ZrO_2(Y)$ ceramic specimens with different porosity during diametral compression tests (a). At the curves, numbers indicate the sample numbers, according to Table 1. (The inset in Fig. 3 (a) shows the deformation curve with a porosity of 42% in the coordinates $\ln(\sigma/\sigma_0) - \ln(\epsilon)$); Dependence of the exponents of strain hardening $n1$ and $n2$ vs. the samples porosity (b)

3. ábra Különböző porozitású $ZrO_2(Y)$ kerámia minták deformációs görbéi a diametrális kompressziós vizsgálatok során (a). A görbéknel a számok az 1. táblázat szerinti mintaszámokat jelölik. (A 3. ábra a) betéte a deformációs görbét 42%-os porozitással mutatja az $\ln(\sigma/\sigma_0) - \ln(\epsilon)$ koordinátákban); Az $n1$ és $n2$ a terhelési keményedés kitevőinek függése a minták porozitásától (b)

The formation of microcracks in ceramics with a porosity of 29% and higher during deformation is caused by the fracture of bridges and lintels of the interpore frame. On the fracture surfaces of ceramics samples with a porosity of 29% and higher, no increase of the monoclinic phase was observed in comparison with the initial state.

The tensile strength (σ_t) decreases with an increase of porosity, Fig. 4 (a). The change of σ_t from porosity is well described by a power function with a high correlation coefficient ($R = 0.99$).

Analysis of the change of the ultimate strain before fracture (ϵ), obtained from the deformation curves « $\sigma - \epsilon$ » (Fig. 3 (a)), showed that the strain (ϵ) decreases slightly from 1 to 0.8% with an increase of the samples porosity (curve 1, Fig. 4 (b)).

The effective modulus of elasticity (E_{eff}), calculated from the slope of the stress-strain curves are decreases with increasing samples porosity (curve 2, Fig. 4 (b)).

Fig. 4 Dependence of tensile strength vs samples porosity (a); Dependence of ultimate strain before fracture (ϵ) (curve 1) and the effective modulus of elasticity (E_{eff}) (curve 2) vs samples porosity (b)

4. ábra A szakítószilárdság függése a minták porozitásától (a); A törés előtti végső fajlagos alakváltozás (ϵ) (1. görbe) és az effektív rugalmassági modulus (E_{eff}) (2. görbe) függése a minták porozitásától (b)

After diametral compression tests of samples with porosity of 4, 17, and 42%, X-ray diffraction analysis was carried out from the front surface of fracture fragments of the samples.

Fig. 5 Macro photographs of samples with different porosity (a - 4%; b - 17%; c - 42%) after diametral compression tests with XRD schemes

5. ábra Különböző porozitású minták makrofotói (a - 4%; b - 17%; c - 42%) XRD sémával végzett diametrális kompressziós tesztek után

The size of the coherently diffracting domains and the crystal lattice microdistortion from different place of the fracture

fragments are determined. X-ray diffraction analysis was carried out for the local points from fractured fragments along the loading axis during diametral compression tests, in the direction starting from the point of contact of the sample with the active platform of the testing machine towards the passive platform with a step of 2-3 mm, the areas for X-ray studies for each point was approximately 4 mm². Fig. 5 (a, b, c) shows macro photographs of samples with different porosities after diametral compression tests with X-ray diffraction analysis schemes.

The dependences of the size of the coherently diffracting domains and the crystal lattice microdistortion of samples with porosities of 4, 17, and 42% are shown in Fig. 6. It follows from the figure that for all samples with different porosity a dispersion both sizes of the CDD and crystal lattice microdistortion are decrease with increase a distance from the active platform of the testing machine and does not exceed 10%. These data indicate the appearances of deformation inhomogeneity of porous ceramics under diametral compression along the compression axis.

Fig. 6 Change of the size of the CDD (a) and the crystal lattice microdistortions (b) of fracture fragments of ceramic samples with porosity of 4, 17 and 42% after diametral compression tests. The start point (zero) is corresponds to the point of contact of the sample with the active platform of the testing machine, and last point (30mm) is corresponding to the point of contact of the sample with the top (passive) platform of the testing machine

6. ábra A kerámiai minták 4, 17 és 42% porozitású töredékeinek CDD méretének (a) és kristálysírkáros mikrotorzulásának (b) változása a diametrális kompressziós vizsgálatok után. A kiindulási pont (0 mm) a minta és a tesztgép aktív platformjának érintkezésénél, az utolsó pont (30 mm) pedig a minta és a tesztelő gép felső (passzív) platformjának érintkezése

4. Conclusions

It has been shown that with an increase of the porosity of ceramic, there are a decrease in the ultimate tensile strength in diametral compression from 115 to 9 MPa and the ultimate

deformation to fracture. The effective modulus of elasticity calculated from the slope of the stress-strain curves also decreases with increasing of porosity.

It been shown that two strain exponent were observed, which indicate a change of deformation mechanism of the ceramic during loading. The decrease of n_2 in ceramics with porosities of 4 and 17% is due to the phase transformation from the tetragonal to the monoclinic phase under the action of applied stresses, and in ceramics with porosity above 29%, it is associated with the formation of multiple microcrack defects during deformation.

It has been shown that in these materials microstructural parameters - coherently diffracting domains of the tetragonal phase and the crystal lattice microdistortions, are changes non-uniformly, which indicate the inhomogeneity of the deformation of this brittle material during compression.

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