

Deformation behaviour of zirconia with a different pore structure

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Abstract. The structure and mechanical properties of zirconia ceramics obtained by slip casting with unimodal and bimodal size distributions and a wide range of porosity (up to 65%) were studied. Samples sintered using powder ZrO_2 -3 mol.% Y_2O_3 with an average particle size of 0.2 μm with an unimodal pore size distribution were obtained by slip casting, followed by removal of the binder. Samples with a bimodal pore size distribution were sintered using spherical ultra-high molecular weight polyethylene particles with an average particle size of 100 μm . Axial and diametral (the Brazilian test) compression tests were performed for the obtained materials. The samples showed a brittle fracture, irrespective of the pore structure in the case of diametral compression tests. In the case of axial compression tests, a typical brittle failure took place in samples with porosity up to 10 vol.% and a “pseudo-plastic” failure occurred in samples with higher porosity. The latter can be ascribed to the movements of the meso-volumes to the pores and the accumulation of multiple damages, which were visually observed during the compression test.

1. Introduction

Mechanical characteristics are one of the key parameters of porous ceramic materials. The deformation behaviour and mechanical properties of porous ceramics are now thoroughly studied mainly for materials with unimodal porosity and a relatively narrow pore size distribution [1, 2]. At present, there is a need to develop materials with a multilevel pore structure, for example, for biomedical applications, when a bimodal pore structure with differing pore sizes is required [3-5]. Nevertheless, studies in which the strength properties and the deformation behaviour of similar materials are simultaneously investigated in a wide range of porosity under different loading schemes are not available in the literature. The aim of this work was to study the mechanical properties of porous zirconia with different pore structures.

2. Materials and research methods

Powder ZrO_2 -3mol% Y_2O_3 with an average particle size of 0.2 μm was used for preparing cylindrical samples with diameter 5 mm and height 7 mm with different porosity types. Samples with an unimodal pore size distribution were obtained by slip casting, followed by removal of the binder. To obtain samples with a bimodal pore size distribution, spherical ultra-high molecular weight polyethylene (UHMWPE) particles with an average particle size of 100 μm in the amount of 25% by volume were added to the slip as a burning pore-forming agent. The samples were sintered in air in the temperature range 1100 – 1600 °C with a sintering time 1 hour. The rate of heating and cooling of the



samples during sintering was 240 °C / h. A metallographic analysis of the polished surfaces of the samples was carried out using an "Altami MET 1M" optical microscope. The pore size distribution was determined from images of polished surfaces of the samples using the ImageJ program. The density and porosity of the samples were determined geometrically. In doing so, the relative standard deviation was less than 5%. The mechanical properties of the sintered samples were measured using the Devotrans universal testing machine. Two different measurements were performed: axial (σ_c) and diametral compression (σ_t) (the Brazilian test). In both cases, the loading rate was 0.2 mm/min, and load-displacement curves were recorded.

3. Results and discussion

Typical images of the structure of the samples and the pore size distributions are shown in figure 1. One can see that the size distributions for small pores were very similar for both types of samples. Large pores were exactly stipulated by the burning pore-forming agent.

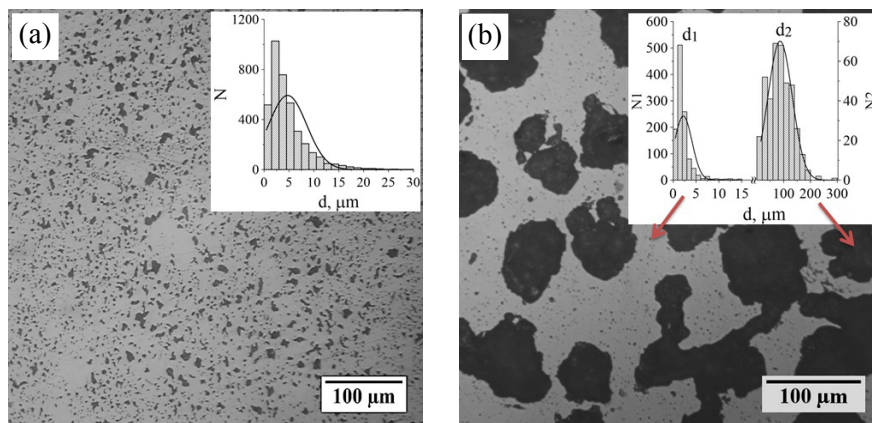


Figure 1. Typical microstructure images of ceramic samples with unimodal (a) and bimodal (b) pore size distributions.

Figure 2 shows the influence of the sintering temperature on the porosity of samples with unimodal and bimodal pore size distributions.

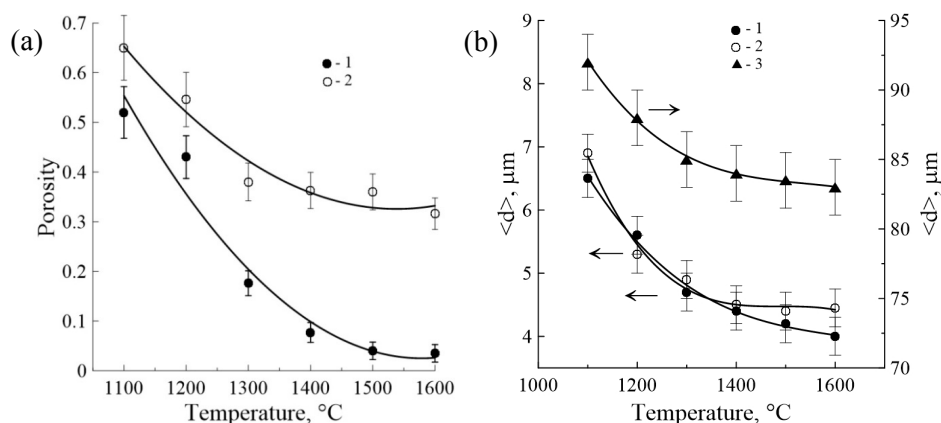


Figure 2. Porosity (a) and mean pore size (b) dependencies on the sintering temperature: 1 - for samples with an unimodal pore size distribution; 2, 3 - for samples with a bimodal pore size distribution.

One can see that with increasing the sintering temperature, the porosity of the samples monotonously decreased, and the porosity values with different types of porosity after sintering at 1400-1600 °C differed by 25-30%. That corresponds to the volume fraction of the introduced pore-forming additive. In addition, with increasing the sintering temperature, the average pore size decreased (figure 2 b). The average grain size in the sintered samples was varied from $d=0.2\ \mu\text{m}$ ($T=1100\ ^\circ\text{C}$) to $d=0.9\ \mu\text{m}$ ($T=1600\ ^\circ\text{C}$).

Figure 3 a represents typical “stress-displacement” curves of samples after diametral compression tests with an unimodal pore size distribution and a porosity of 3.5% and a bimodal pore size distribution and a porosity of 38%. Comparison of the “stress-displacement” curves after diametral compression tests shows that the fracture type of the samples did not depend on the type of porosity and the pore volume. All samples were characterized by elastic behaviour. On the active loading stage, sharp changes of the stress value were observed, which can be ascribed to the formation of microcracks. This did not lead to the macrofracture of samples, followed by a brittle fracture.

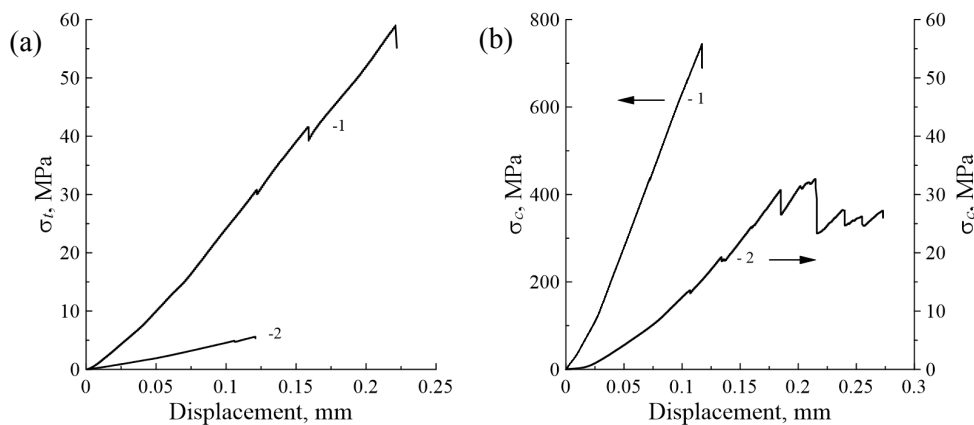


Figure 3. Typical “stress-displacement” curves of zirconia ceramic samples obtained: a - after diametral compression tests with a unimodal pore size distribution and a porosity of 3.5% (curve 1) and a bimodal pore size distribution and a porosity of 38% (curve 2); b - after axial compression tests with an unimodal pore size distribution and a porosity of 3.5% (curve 1) and a bimodal pore size distribution and a porosity of 53% (curve 2).

Figure 3 b represents typical “stress-displacement” curves of samples after axial compression tests with an unimodal pore size distribution and a porosity of 3.5% and with a bimodal pore size distribution and a porosity of 53%. Comparison of “stress-displacement” curves after axial compression tests shows that the fracture type of the samples depended on the type of porosity and the pore volume. The samples with an unimodal pore size distribution and a porosity up to 10% were characterized by elastic behaviour and brittle fracture (curve 1), whereas the samples with a bimodal pore size distribution and high porosity demonstrated saw-tooth oscillations of the stress value after reaching the ultimate compressive strength indicating accumulation of damages. Thus, under the axial compression test, a transition from typical brittle fracture in samples with porosity up to 10 vol.% to “pseudo-plastic” fracture in samples with a higher porosity took place regardless of the type of pore structure. This behaviour is typical of highly porous ceramic materials and is associated with the appearance and accumulation of multiple damages [6-9], which can be visually observed during the compression test.

Figure 4 shows the dependences of ultimate compressive and ultimate strength for diametral and axial compression samples with unimodal and bimodal pore size distributions vs. porosity. One can see that with increasing porosity, the ultimate strength decreased, and these dependences are well described by an exponential equation of the type:

$$\sigma = \sigma_0 \exp(-bP) \quad (1),$$

where σ is the strength of a porous material, σ_0 is the strength of a material free of pores, P is the volume fraction of pores and b is a coefficient, which depends on the structure and composition of the material [10]. The analysis of the obtained dependences showed that the coefficient b was 6-7 for all dependences that is consistent with the literature data [10].

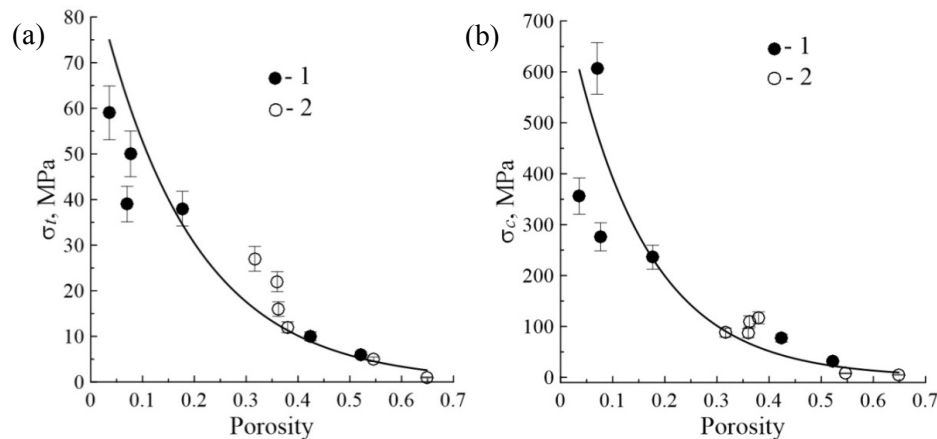


Figure 4. Dependences of the ultimate strength for diametral compression (a) and axial compression (b) on porosity obtained for zirconia samples with unimodal (1) and bimodal (2) porosity.

4. Conclusions

Zirconia ceramics samples with different types of the pore structure demonstrated a brittle fracture, and only single microcracks were formed on the active stage of loading. For the zirconia ceramics samples having high porosity and subjected to axial compression tests, the accumulation of microdamages occurred without fracture of the entire sample. All dependences of the ultimate strength on porosity were well described by an exponential equation with exponent factors in the range of 6-7.

Acknowledgments

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