Investigation of mechanical properties of porous alumina ceramics: experiment and simulation

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Abstract. The paper presents the experimental data on elastic and strength characteristics of specimens made of porous Al₂O₃ ceramics in three-point bending. Ceramics specimens were prepared by the slurry casting of commercially pure Al₂O₃ powder and subsequent sintering at the temperatures 1400, 1500, and 1600 °C. The simulation results were reported for the mechanical behaviour of specimens of porous Al₂O₃-based ceramics at the mesolevel. We studied the mesovolumes with an explicit treatment of pores loaded in uniaxial tension. The pore structure of the mesovolumes was borrowed from experimental SEM images. The mesoscopic fracture was described using the fracture criterion which includes the Lode parameter to make allowance for the stress state type. Despite wide scatter, the experimental and calculated elastic and strength characteristics of the material agree rather well.

1. Introduction
Now porous ceramic materials are of interest for different technical applications, e.g., filters, membranes, insulators or biomedical implants. Porosity is one of the main microstructural parameters for ceramics. The effect of porosity on the variation of elastic and strength properties of porous materials have been studied by numerous researchers, using various mathematical models for describing inelastic deformation and fracture of these materials as well as different experimental techniques [1–5]. Wide use has been made of models of physical mesomechanics for the numerical study of the deformation and fracture features and to predict the mechanical behaviour of heterogeneous materials at different length scales [6–13].

The uniaxial tensile test for the brittle material is impossible because of their traits. Therefore the three-point bending flexural test is used to assess the mechanical characteristics of these materials intensity. This method provides values for the modulus of elasticity in bending and flexural stress of the material.

In this study, the experimentally and numerically properties of the Al₂O₃-based porous ceramic specimens in three-point bending was investigated. The rectangular specimens with various values of porosity ranging from 17 to 33 % were chosen for the bending test.

2. The results of experimental investigation
Ceramic specimens were made by the slurry casting of commercially pure Al₂O₃ powder and subsequent sintering at the three temperatures 1400, 1500, and 1600 °C during an hour. The produced specimens had the form of rectangular parallelepipeds. The initial dimensions of the specimens were 65×7×5 mm. The specimens were tested in three-point bending to determine their mechanical
properties. The porosity of specimens was 33, 26, and 17 % for the sintering temperatures 1400, 1500, and 1600 °C, respectively. The Image J program was used to determine the porosity of specimens. SEM images of the surface of Al₂O₃ specimens are shown in Figure 1.

![SEM images of the etched surface of Al₂O₃ specimens sintered at the temperatures 1400 (a), 1500 (b) and 1600 °C (c)](image)

The ultimate strength and elastic modulus of ceramic specimens were determined in static three-point bending at the loading rate of 1 mm/min. Five specimens of the same batch were taken. The load-displacement curves were employed to evaluate the mechanical properties of the ceramic specimens. Using experimental data the values of the ultimate strength (σ_{exp}) and elastic modulus (E_{exp}) were calculated by the following equations:

\[ \sigma_{exp} = \frac{3PL}{2bh^2}, \quad E_{exp} = \frac{l^3m}{4bh^3} \]

where \( P \) is the maximum load, (N); \( l \) is the support span, (mm); \( b \) is the width of the specimen, (mm); \( h \) is the thickness of the specimen, (mm); \( m \) is the gradient (i.e., slope) of the initial straight-line portion of the load-deflection curve, (N/mm). The calculated values of the ultimate strength and elastic modulus are presented in table 1.

| Sintering temperature, °C | Porosity, %  | \( \sigma_{exp} \), MPa | \( E_{exp} \), GPa | \( E_{calc} \), GPa | \( \sigma_{calc} \), MPa |
|--------------------------|--------------|-------------------------|-------------------|------------------|-------------------------|
| 1400                     | 33±0.7       | 138±27                  | 58±7              | 35±9             | 150±13                 |
| 1500                     | 26±0.6       | 243±14                  | 80±13             | 85±23            | 236±7                  |
| 1600                     | 17±1         | 265±35                  | 113±16            | 206±9            | 286±17                 |

3. Simulation results and discussion

In three-point bending one of the parts of the specimen is in the tensile state, being most dangerous for porous ceramics. Consequently, uniaxial tension of mesovolumes with an explicit treatment of pores is modeled at the mesolevel in this work. Porous structures of the mesovolumes were borrowed from the electron microscopy data for the studied specimens (figure 2). Dimensions of mesovolumes in figure 2a and 2b are 100×100 μm. Three different digital mesovolume models of the same porosity were chosen for each sintering temperature.

The simulations were performed for problems in a two-dimensional formulation of plane-strain conditions using the finite difference method. We made use of constitutive equations that take into account the damage accumulation, which causes degradation of elastic properties [8]. The damage accumulation kinetic equation is built upon the effective stress by the Drucker-Prager material model and includes the Lode parameter to make allowance for the stress state type. To represent the
mesoscopic fracture we took the critical damage criterion. Since the fracture criterion is fulfilled, the stresses are reduced to zero, and then the material stops to resist tension but not compression.

![Figure 2](image1.png)

**Figure 2.** Computer models of the ceramic structure with the porosity (a) 26 % and (b) 17 %

The results of the simulation at the mesolevel are shown in figure 3. The stress-strain curves averaged over the mesovolume are depicted in figure 3a. They appear as characteristic ones for brittle materials. We can notice that an increase in sintering temperature causes nonlinear growth of Young's modulus and strength of the porous material. Note, that the higher sintering temperature, the lower porosity. The calculated values of the elastic modulus ($E_{calc}$) and strength ($\sigma_{calc}$) are presented in table 1. It can be noted that the calculated strength values agree well the experimental ones. The calculated Young's modulus fits the experiment only for the sintering temperature 1500 °C, while it is lower than the corresponding experimental value at the sintering temperature 1400 °C and is higher than the experimental value at the 1600 °C. We mention that in general, the calculated elastic modulus and strength values roughly agree with the experimental data for the corresponding porosity [1].

Figures 3b and 3c display the fracture patterns in the two mesovolumes. One can see the cracks propagated from the potent stress concentrators specified by the pore shape and arrangement. The orientations of the cracks are mostly perpendicular to the tensile loading axis, which is typical for brittle materials fracturing.

![Figure 3](image2.png)

**Figure 3.** Mesoscale modeling results: (a) averaged stress-strain curves and fracturing patterns in mesovolumes of porous ceramics with the porosity (b) 25 % and (c) 17 %

4. Conclusion
Using the experimental results of the three-point bending test, we deduced the values of elastic modulus and tensile strength of the alumina ceramic specimens with the pore content varying from 17 to 33%. The experimental results evidence a decrease of the porosity with sintering temperature
increasing, while elastic moduli and tensile strength increase nonlinearly. We mention in passing the wide scatter (in the range of 12–20%) of the experimental data for both Young's modulus and strength, yet the dispersion of porosity does not exceed 2–6% for each of the sintering temperatures. This is indicative of the profound effect of diverse parameters exemplifying the pore structure on the mechanical properties of porous materials (as also noted elsewhere [14]).

Here, we have analyzed how porous structure impacts on the features of mesoscopic fracturing and macroscopic stress-strain curves of the porous ceramics using our simulation results at the mesoscale. To perform modeling for the same total porosity, we used three different mesovolumes that vary in the pore shape and location specified by experimental photographs. The results obtained indicate that potent stress concentrators available in the mesovolumes determine the fracture nucleation site and influence the crack propagation. For all sintering temperatures, the calculated values of strength agree closely with experimental ones, while the calculated values of elastic modulus are distinct from the experimental ones at the sintering temperatures 1400 and 1600 °C, despite relatively large dispersion. The scatter of the calculated values is conditioned by the availability of heterogeneities of different shape and arrangement in the mesovolumes.

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