Abnormal Behavior of ZrO_2-MgO Porous Ceramic Composite under Compression

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Abstract. In this work ZrO_2-MgO porous ceramic composite fine crystalline structure was studied, its microstructure and mechanical properties after sintering at 1600°C. Pores had a bimodal size distribution with the mean sizes of about 30 and 100 μm. It was shown that the introduction of organic pore-forming particles into the initial powder composition of ZrO_2-MgO allows producing ceramics with a bimodal pore structure, the material strength is mainly determined by microstresses, and in general, such porous ceramics show uncharacteristic behavior under compression, which is confirmed by plot of the Hall–Petch dependence of microstresses on the average crystallite size with the coefficient $K = 0.38$ MPa $m^{1/2}$, corresponding to plastic materials.

INTRODUCTION

Improving the effectiveness of materials' functional properties is one of the main tasks of modern material science. In recent years, actively investigated technologies of materials micro- and macrostructure varying, determine their possible uses. For example, it is proved that creating a porous structure allows improving such parameters as filtering ability or heat resistance of ceramic materials [1, 2]. In the field of osteoimplantology one of the most important characteristics of porous materials is the morphology of pores and their total volume and it has already been shown that the most favorable material for osteointegration is a ceramic material with a bimodal pore structure and average pore size about 100–500 and 10–20 μm.

A special role among porous composite materials belongs to ceramics due to their high mechanical parameters, corrosion and wear resistance. Porous composite based on zirconium and magnesium oxides is of a special interest as an osteoreplacement material because of its biocompatibility and osteoconductivity [3].

In [4, 5], the structure and properties of a ZrO_2-MgO ceramic composite with a MgO concentration up to 43 mol % were studied, and it was shown that with cubic ZrO_2 concentration increasing the compressive strength decreases. Increasing in MgO concentration leads to the growth of cubic ZrO_2 grain size, characterized by low strength and fracture toughness, but at the same time increasing in MgO concentration results in the strength increase.

However, at the present time there is a lack of scientific knowledge about the behavior of such oxides system in a wider range of compositions. In the previous work a porous ceramic composite was already obtained and the morphology of its structure was studied [6]. Studying ceramic composite ZrO_2-MgO with bimodal porosity in a wider range of components concentrations will allow determining the influence of composition, micro- and fine crystal structure parameters on the strength of this material and possibilities of its application in the field of osteoimplantology and bone replacement where the similarity of the implant and bone tissue strength parameters is one of the main conditions.

The aim of this study is to investigate the dependencies between micro- and fine crystal structure, strength and composition of ZrO_2-MgO ceramics with bimodal porous structure.

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MATERIALS AND METHODS

In this paper is devoted to studying the porous ceramic materials ZrO_2-MgO, obtained by cold uniaxial compaction with a hydraulic press under a pressure of 180 MPa of zirconia powder with a particle size of 0.5–6.0 μm and magnesium oxide (MgO), 2 μm average particle size, mixtures with a concentration of 0, 25, 50, 75 and 100 mass % and sintering at a temperature of 1600°C. The porosity of ceramics was obtained by organic pore-forming particles with an average size of 100 μm adding to the initial powder mixtures.

Pore morphological structure was studied by scanning electron microscope Tescan VEGA 3 of ceramics polished internal surfaces and pores distribution was calculated by the method of random secants. The strength parameters were studied by universal testing machine Devotrans DT with a constant rate of compression of 0.1 mm/s.

X-ray diffraction studies using X-ray diffractometer DRON-3 in the angular range of 2θ from 20° to 120° allowed determining the dependences between the content of MgO and crystallites average size (coherent diffraction domains (CDD), microdistortions using Hall–Williamson equation.

RESULTS AND DISCUSSION

In Table 1 the results of studying pore microstructure are shown. Pores are presented by bimodal size distribution with the average pore size of about 30 and 100 μm. This structure is caused by burnout of the pore-forming particles, whose average size corresponds to the size of large pores, and the packing of ceramic powder particles, between which smaller pores arrange. It is found that the pore volume, average size of pores and standard deviation do not depend on the composition.

On Fig. 1 the average crystallite size and crystal structure microdistortions of composite are shown. Crystallite sizes of ZrO_2 cubic phase increase linearly with increasing of MgO concentration, at the same time microdistortions of ZrO_2 decrease with a concentration of MgO from 0 up to 25 mass % and do not change with a further increasing of MgO concentration. The average crystallite size of MgO decreases with an increase in its concentration up to 50% and does not change after that. MgO microdistortions linearly decrease with increasing in MgO content in composite.

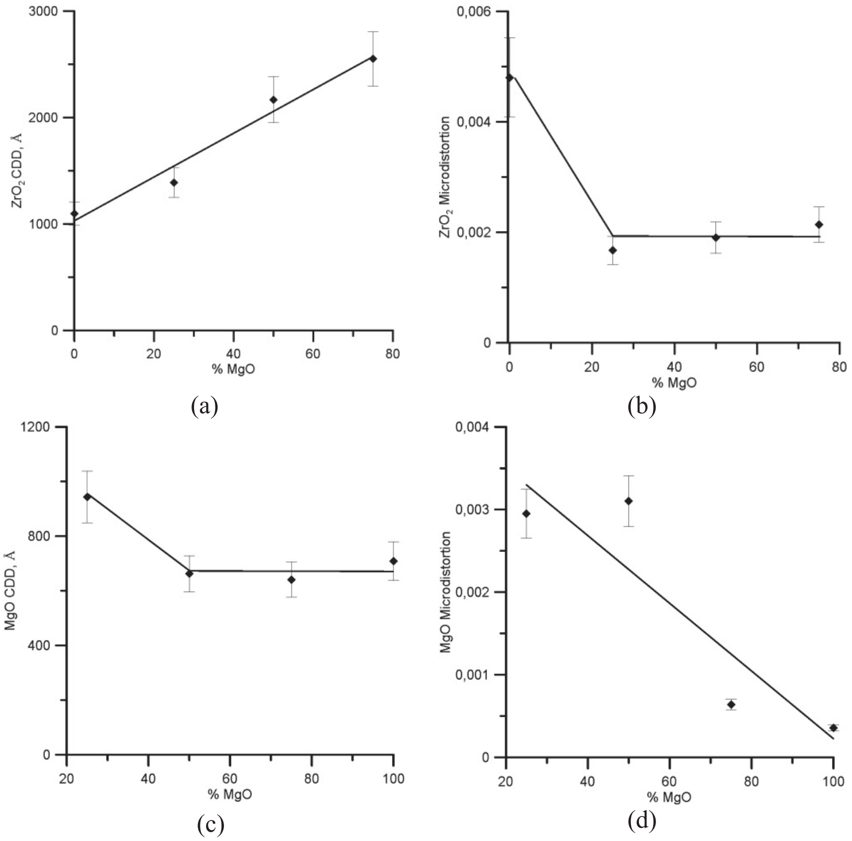
The analysis of studied ceramic samples stress-strain diagrams showed that the composite compressive strength varies from 33 to 18 MPa.

X-ray diffraction studies allowed obtaining an average value of the composite crystal lattice microdistortions, calculated by the mixture rule. Multiplying these microdistortions by the composite theoretical elasticity modulus, also calculated by the mixture rule, makes it possible to estimate the microstresses in crystallites. On Fig. 2 the dependence between macro compressive strength and microstresses is shown and it can be seen that with achieving 25 mass % of MgO concentration microstresses sharply decrease and almost do not change with further increasing in MgO amount. This dependence has an exponential-like shape. It is seen that the composite strength is completely determined by the value of inner microstresses.

According to these data a dependence between the internal microstresses and average crystallites size of composite, calculated by the mixture rule, was plotted (Fig. 3). This dependence has a linear form, which corresponds to the Hall–Petch equation. In this work coefficient K is equal to 0.38 MPa $m^{1/2}$, this value is close to Lead K coefficient (K_{Pb} = 0.33–0.43 MPa $m^{1/2}$) [7] and corresponds to the plastic materials behavior. In [8] authors studied dense ceramic composite ZrO_2-MgO, and found, that Hall–Petch coefficient K was equal to 0.74 MPa $m^{1/2}$, which corresponds to brittle materials.

TABLE 1. The average size of pores and pore volume of studied ceramics.

ZrO_2, mass %	Average pore size of the small pores, μm. Standard deviation	Average pore size of the large pores, μm. Standard deviation	Porosity, vol %
100	29 ± 19	110 ± 31	45
75	30 ± 23	104 ± 21	43
50	27 ± 17	94 ± 27	45
25	26 ± 17	101 ± 30	49
0	28 ± 20	105 ± 27	47



(a) (b)

(c) (d)

FIGURE 1. Dependences of crystalline lattice CDD and microdistortions of the composition: (a) CDD ZrO_2; (b) microdistortions of ZrO_2 cubic crystallite lattice; (c) CDD MgO; (d) microdistortions of MgO crystalline lattice

Such anomalous behavior under axial loading of porous ceramics, which is usually characterized by high hardness and very brittle fracture, can be explained by pseudo-elastic behavior and this deformation effect may correspond to the high-pore content with an average volume of 50%, micromechanical instability and possible movements of material fragments due to the pressure effect of porous ceramic composite [9, 10].

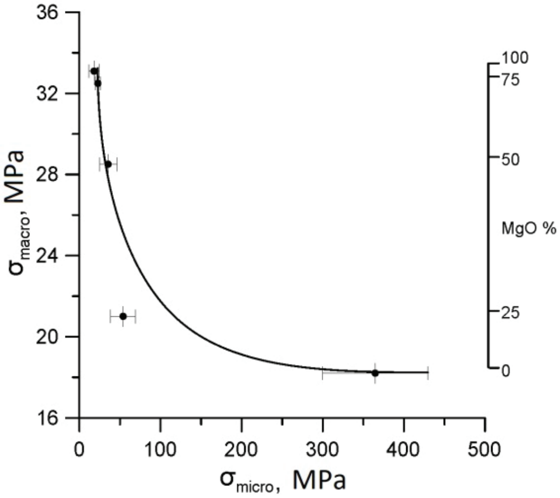


FIGURE 2. Dependence of material tensile strength from microstresses of composite, calculated by the mixtures rule

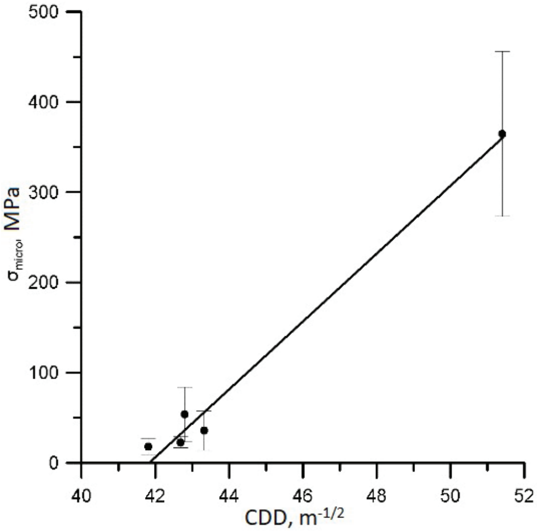


FIGURE 3. Hall–Petch dependence of microstresses from CDD in composite

CONCLUSIONS

It has been shown that the macro-strength of porous ZrO_2-MgO composite ceramic is completely determined by internal microstresses, and the increase in micro-stresses leads to decreasing in the compressing strength.

It was found that in porous ZrO_2-MgO composite ceramic there is Hall–Petch-type dependence between micro-stresses vs. average crystallite size with Hall–Petch coefficient K = 0.38 MPa $m^{1/2}$, which corresponds to plastic materials and is two times lower than for the same ceramic composite without porosity. This anomalous value of Hall–Petch coefficient may be explained by pseudo-elastic behavior under deformation of high-pore brittle material, micro-mechanical instability and possible movements of material fragments due to the pressure effect of porous ceramic composite.

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