Investigation of ZrO_2 Nanoceramics Microstructure

O.V.Karban[1], O.L.Khasanov[2]

[1] Physicotechnical Institute UrB of RAS, 132 Kirova st., 426001 Izhevsk, Russia
e-mail: uds@pti.udm.ru phone: (3412)212655 fax: (3412)250614
[2] Tomsk Polytechnic University, 30, Lenin Ave., 634050 Tomsk, Russia
e-mail: khasanov@tpu.ru phone: (3822)427242 fax: (3822)427242

It is well known that the technological methods used for preparing nanoceramics, influence both the structure and properties of created materials. An efficient method of creating compact nanoceramics is known to be dry pressing of ceramic nanopowders under simultaneous ultrasonic (US) treatment [1]. The latter provides uniform dense green compacts. Besides, the nanostructure of a powder retains unchanged here in the process of its compacting and sintering.

The focus of the present work is given to the investigation of the structure formation and characteristics of yttrium-stabilized zirconium dioxide ceramics, sintered by uniaxial pressing under US-vibrations of varying power (0÷3 kW) and compaction pressure (P=50÷200 MPa).

Experimental and specimens

Nanoceramics were sintered from green compacts of ZrO_2 –5%wtY_2O_3 nanopowder, produced by the plasmochemical synthesis [1]. The powder particles had the shape of polycrystalline plates (scaly morphology) with the average linear size of 150 nm; the mean crystallite size in such a plate was 23 nm.

To compact powders, techniques described in [1] were used. The resulting compacts were sintered in air at T_s = 1650°C. Details of XRD, SEM and EPMA analysis, mechanical properties of ceramic samples were investigated by techniques, references of [1]. The AFM-measurements were performed in tapping mode using a scanning probe microscope P47-MDT (NT-MTD, Russia). The samples were newly spalled.

According to EPMA, the initial powder contained 74.13% Zr, 18.16% O, 4.28% Y and 3.43% of admixed carbon was also present. All the samples were polycrystalline tetragonal zirconium dioxide (Y-TZP). The crystallite mean size $<d>$, according to XRD, the mean grain size and physico-mechanical characteristics are given on Fig.1 as a function of compacting conditions.

Experimental results and discussion

It is widely known [2] that the decrease of crystallite (grain) sizes to as low as nanometric-scaled ones (d >12-20 nm) gives rise in strength, hardness and plasticity of ceramic materials. It is seen from the XRD and SEM-data that the dependence $<d>$ and D_{gr} of the sintered nanoceramics on the US-power W and compacting pressure P is not monotonic (Fig.1). It also does not correlate with such physico-mechanical properties (Fig.1) as microhardness H_v, bending strength σ_b and fracture toughness K_{Ic}. The absence of a traditional correlation between microstructural characteristics and macroscopic mechanical properties is indicative of the leading role of intergrain interfaces in the nanoceramics under study. It should be taken into account that while manufacturing nanoceramics a number of uncontrolled factors (temperature gradients, different levels of grain defectness after US-activation during compacting, etc.) can result in a different quality of intergrain bonds.

The objective of the present work is to comparatively analyze the structure of the ceramics, sintered from green compacts after a conventional static dry compacting (O-ceramics) and after compacting nanopowders under ultrasound (US-ceramics).

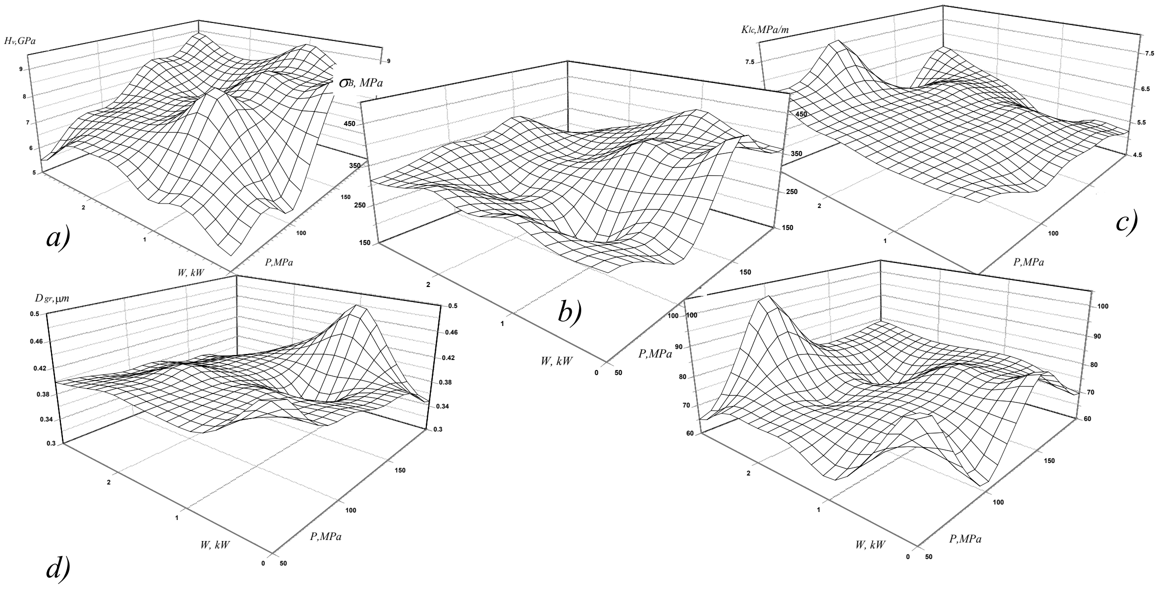


Fig.1 *Microhardness H_v (a), bending strength σ_b (b), fracture toughness K_{lc} (c), mean grain size D_{gr} (d) and crystallite mean size <d> (e) vs US-vibrations power W and pressure P*

The main distinction found between these is their differing trend in grain size vs. compacting pressure dependences. Also, a small quantity of the interfacial phase is observed in the US-ceramics. It is typical of the O-ceramics (Fig.2 *a,b*) to demonstrate a monotonic drop in grain sizes with pressure. With the US-ceramics (Fig.2 *c,d*), the dependence of grain sizes on pressure is non-monotone. All samples preserve the plate-scale structure of grains with their thickness of 20-100 nm. However, in the O-ceramics, the scaly structure becomes less pronounced with the increase of *P*. Apparently, plastic deformation results in the fusion of scales. The sonication of samples enables a lower rate of plastic deformation of particles in a compact. At the same time, the particle structure varies due to mechanoactivation.

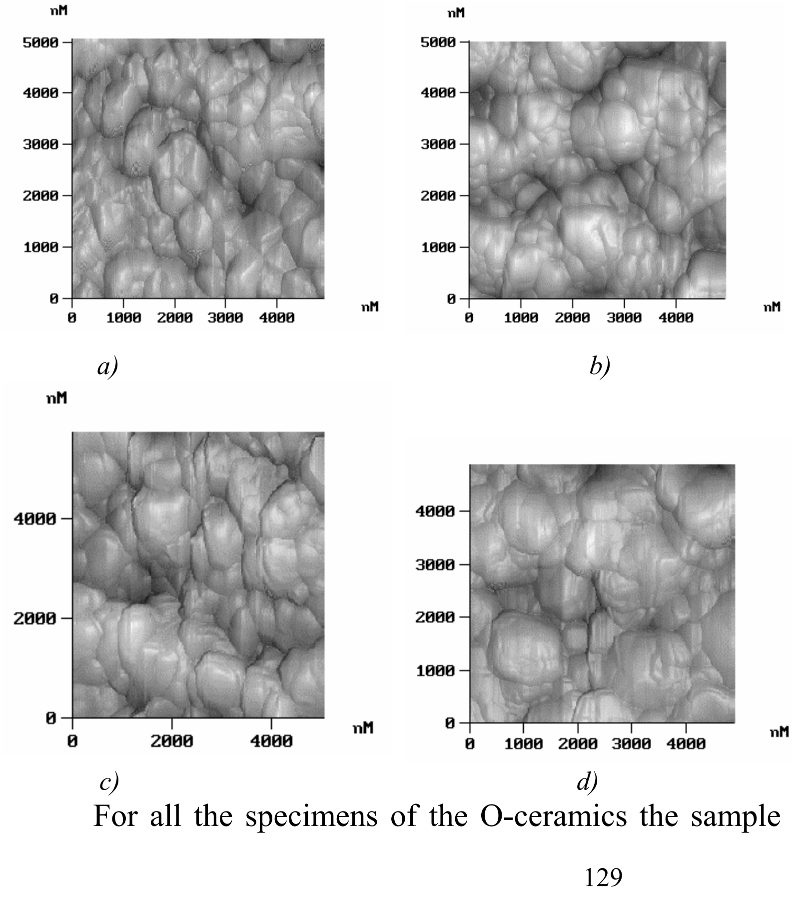
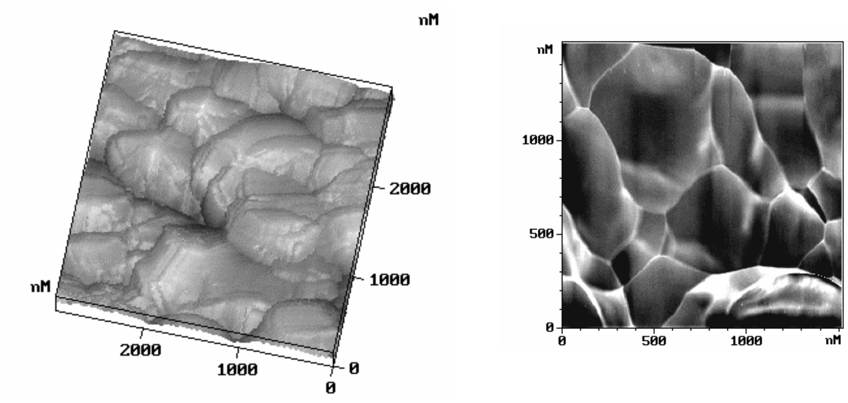


Fig.2. *Topography image for O-ceramics (a-50 MPa, b-200 MPa) and US-ceramics (W = 3kW; c-50 MPa,d-200 MPa)*

For all the specimens of the O-ceramics the sample breakdown occurs involving an intercrystalline mechanism, which points to the instability of grain boundaries. In the case of the sonicated ceramics, the transcrystalline mechanism of material breakdown is sometimes observed (Fig. 3 *a*) along with the decreased bending strength. The increase in the US power up to 3 kW is enough to additional activation of diffusion during sintering, which can result in the segregation of elements (admixtures, for example) at grain interfaces (Fig.3 *b*). The thickness of this segregated phase is 4÷6 nm. Its elastic characteristics are different from those of the matrix. This is especially apparent in the samples obtained at 2 kW –150 MPa, 3 kW - 100 and 200MPa. The emergence of this segregated phase results in the stabilization of grain boundaries The interfacial regions, that are chemically different from the matrix due to the US-induced segregation of the admixture, can serve as a dispersion-strengthening phase and prevent the propagation of cracks (Fig.1*c*). The composition of the interfaces can be determined by means of additional investigation methods, such as XPS, for example.



a) *b)*

Fig.3. Topography image(a) for samples (W =1kW, P = 100 MPa) and phase contrast image for samples (W =3kW, P = 200 MPa)

The investigations performed evidence the essential influence of the intergrain boundary structure on the physico-mechanical properties of nanoceramics. The perfection of the internal grain structure and the optimization of the grain interface structure - these are the ways to improve strength characteristics of nanomaterials.

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