AFM, XPS and X-ray investigations of nanoceramics.

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Great interest in nanocrystalline materials is due to excellent service characteristics of these materials resulting from both the size effect and structural changes in interfacial regions. The present work is devoted to the investigation of processes that take place in nanoceramics under baking.

Experimental and specimens

Nanoceramics were obtained from Al_2O_3 powder (0.5δ+0.5γ) doped with 1.5%MgO_2 (the mean grain size of 20-30 nm) by magnetic-pulse compacting [1] with subsequent baking at temperatures < 1450°C (Table).

The measurements were performed using a scanning probe microscope *P4-SPM-MDT, P47-MDT* (NT-MTD, Russia) and an *EC-2401* spectrometer. The samples were newly spalled.

№	*T_{baking}, °C*	*τ, (min)*	*Phase Bec.%*	*d_{grain} (X-ray),nm*	*$D_{particle}$, nm*	*d_{ACM}, nm*	*Roughness, R_q nm*
1	—	—	γ-50, δ-50		200-300	25	71.6
2	1100	—	γ-76,δ-24 α-0.5	23	200-300	45	43.5
3	1170	—	γ-68,δ-11 α-2.1	40	250-350	80-120	39.2
4	1260	—	α-96 $MgAlO_3$ - 4	117	250-400	120	44.1
5	1310	—	α-96 $MgAlO_3$ -4	164	250-400	35	58.3
6	1370	—	α-96 $MgAlO_3$ -4	189	250-400	12	67.7
7	1410	2	α-96 $MgAlO_3$ -4	300	600	35*	70.3
8	1410	20	α-96 $MgAlO_3$ -4		450-650	35*	85.3
9	1410	60	α-96 $MgAlO_3$ -4	1000	1000-1500	35*	124.5

Experimental results and discussion

The results of the studies performed allowed all samples to be divided into two groups: ceramics baked at temperatures below and above 1400°C.

<u>In the first group</u> only the grain growth is observed and the particle size (300-450 nm) remains unchanged as compared to the original powder.

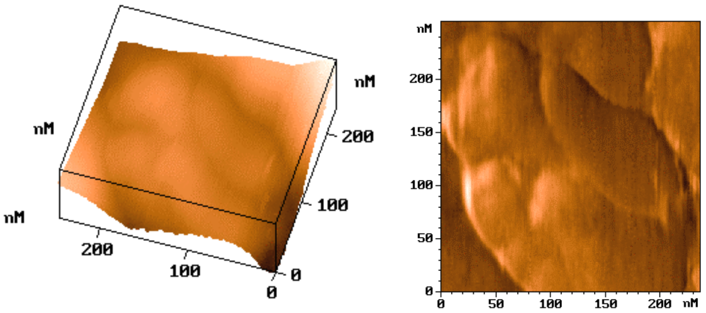

Fig.1. ***3D-AFM image and 2D-LFM image for sample 2***

For the ceramics baked at temperatures below 1170°C (Fig.1) the structure of particle surface coincides with that of the original powder, and the γ-phase becomes dominant. The grain size observed from the AFM experiment exceeds the X-ray value (Table). The effect observed seems to be a purely surface phenomenon due to the phase transition of γ-Al_2O_3 =>α-Al_2O_3 at the particle surface at baking temperatures > 1000°C [2]. This process is accompanied by a decrease in the specific surface and roughness (Fig.2).

The XPS data on the Al and O concentration for specimen 1 (Fig.3) suggest a recrystallization of the particle surface on exposure to the ion beam under etching. The chemical activity and the specific surface decrease, which results in the decrease of its adsorptivity and the reduction of O content. The roughness of specimen 1 after ion etching (R_q = 46 nm) is comparable with that of specimens 2 and 3 (Table).

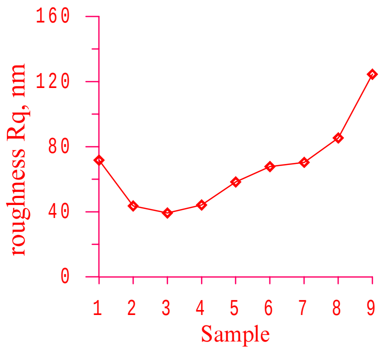

Fig.2 ***Roughness vs number of sample***

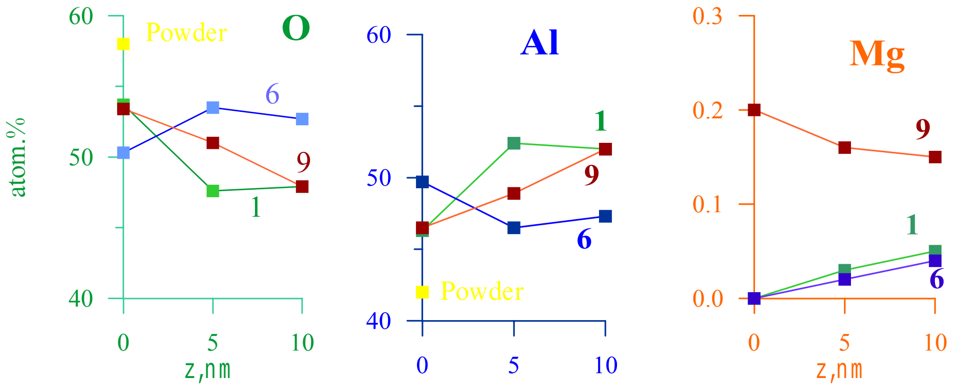

Fig.3 ***Al, O and Mg content for specimen 1, 6, 9 under ion etching***

Increasing the baking temperature above 1230°C (Fig.4) results in complete transformation of γ-Al_2O_3 into the α-modification and further grain growth. A structure of size 10-35 nm revealed in the interfacial region by AFM investigation (Fig.4) is probably a result of initiated melting of the particle surface at temperatures > 0.6 T_{melt} of the bulk specimen, which leads to an increase in roughness (Fig.2).

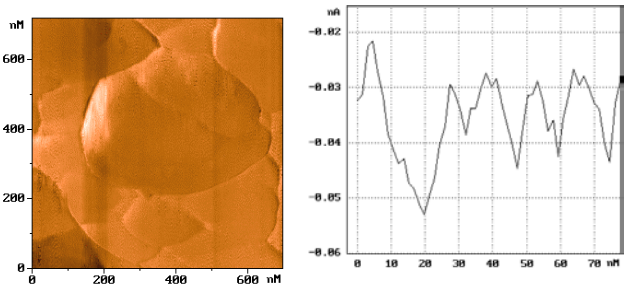

Fig.4 ***2D-LFM image and cross-section for sample 6***

According to the XPS data, the Al and O content for specimen 6 remains practically unchanged under ion etching, and the Mg content in the interfacial region is minimum (Fig.3). This points to the initiated stabilization of the interfacial regions However, the baking temperature and. duration are not sufficient to complete this process. For all specimens of the first group the sample breakdown occurs by intercrystalline mechanism, which points to the instability of particle boundaries.

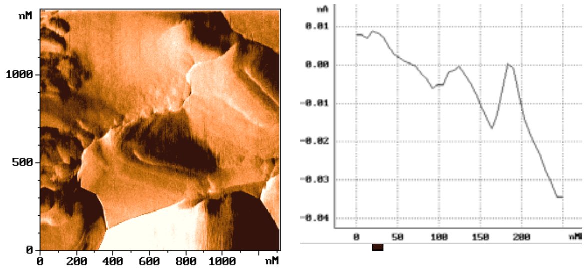

Fig.5 ***2D-LFM image and cross-section for sample 9***

For the second group the grain size coincides with the average particle size. The particle size grows from 600 nm(baking time τ=2 min.) to 1500 nm (τ=60 min.) in consequence of secondary recrystallization. The nature of breakdown changes from intercrystalline to transcrystalline. For specimen 7 (τ=2 min.) there appear individual particles with transcrystalline breakdown. For specimen 8 (20 min), such breakdowns amount to about 50%, whereas for specimen 9 (60 min) only transcrystalline breakdown is observed (Fig.5), which suggests the stabilization of the interfacial regions. A structure 25-35 nm in size detected in the lateral force mode for the second-group samples indicates the brittle character of breakbown **.**

In this group of specimens the Mg content changes weakly with the thickness of the analyzed layer (Fig.3). The changes in the Al and O content (Fig.3) are indicative of high activity of the breakdown surface and indirectly attest the transcrystalline mechanism of breakdown.

Conclusions

The investigations performed have shown that the use of atomic force microscopy (AFM) and XPS methods along with traditional X-ray diffraction makes it possible not only to examine the structural changes in nanomaterials and the integral grain size but to obtain additional information on topographical and structural features of nanoparticles and interfacial regions.

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