Dimensional and phase characterization of Al_2O_3 nanoparticles obtained by LEC technique

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The synthesis and structural investigation of nanoparticles of various materials with well-controlled narrow size distribution are major goals in the development of future technologies and metrological support of nanotechnology. The basic powder characteristics such as size, shape, state of agglomeration, surface chemistry and crystal phase composition depend on the method of preparation. Knowledge of the lifecycle risks of these properties of nanomaterials for the environment, health and safety is of great interest and importance.

Aluminum oxide nanoparticles can be prepared by a wide range of methods and have a lot of applications in the field of material science, microelectronics industry, medical science and engineering. Pulsed laser ablation is known to prepare high purity different material nanoparticles with narrow size distribution and shape close to spherical. The most important quality of obtained powders is low agglomeration of nanoparticles. The specific surface area is equal to 56 m^2/g, which corresponds to average surface-volumetric effective particle diameter d_{BET} = 14.8 nm.

In the present study nanoparticles were synthesized by target laser ablation (LEC) [1]. The targets were prepared from micron size Al_2O_3 powder.

Obtained Al_2O_3 powder was investigated by high resolution transmission electron microscopy, electron diffraction and X-ray diffraction. TEM, HRTEM and ED were performed on a FEI Tecnai G^2 12 at 120 kV and a FEI Titan 80-300 at 300 kV. X-ray powder pattern was obtained at BELOK station (Kurchatov Synchrotron Centre) with CCD 2D-area detector, λ = 0.9845 Å.

At low magnification Al_2O_3 nanoparticles have almost spherical shape (Fig. 1a). The diameters of 1477 particles are measured and distribution diagram is shown in Fig. 1b. The diagram has its maximum at 24 nm. Along with this there are particles with average diameter about 120 nm (32 nanoparticles are measured).

HRTEM profile images reveal atomic steps on the spherical nanoparticles surface (Fig. 2a). The step size is equal to 2-3 space distances. Sometimes thin 2-3 nm amorphous layer is observed which obviously appear after e-beam radiation (Fig. 2b).

Lattice parameters are investigated by electron diffraction and Fast Fourier transforms. It is possible to conclude that nanoparticles with cubic lattice are of 7-40 nm in size and nanoparticles with orthorombic lattice more often are larger (more than 40 nm).

Bragg peak parameters were measured by pseudo-Voigt function fitting. Two phases with cubic (Fd-3m, a = 7.906(3) Å) [2] and orthorhombic (P222, a = 7.92(1) Å) [3] lattice were revealed, the cubic phase being prevalent (Fig. 3).

In summary, the investigated nanosized aluminum oxide particles have nearly spherical shape regardless of size, narrow distribution, they are monocrystalline and mostly in cubic phase. Also with a decrease in size from 40 to 10 nm instead of the orthorhombic δ-Al_2O_3 structure the cubic Al_2O_3 phase is observed.

References

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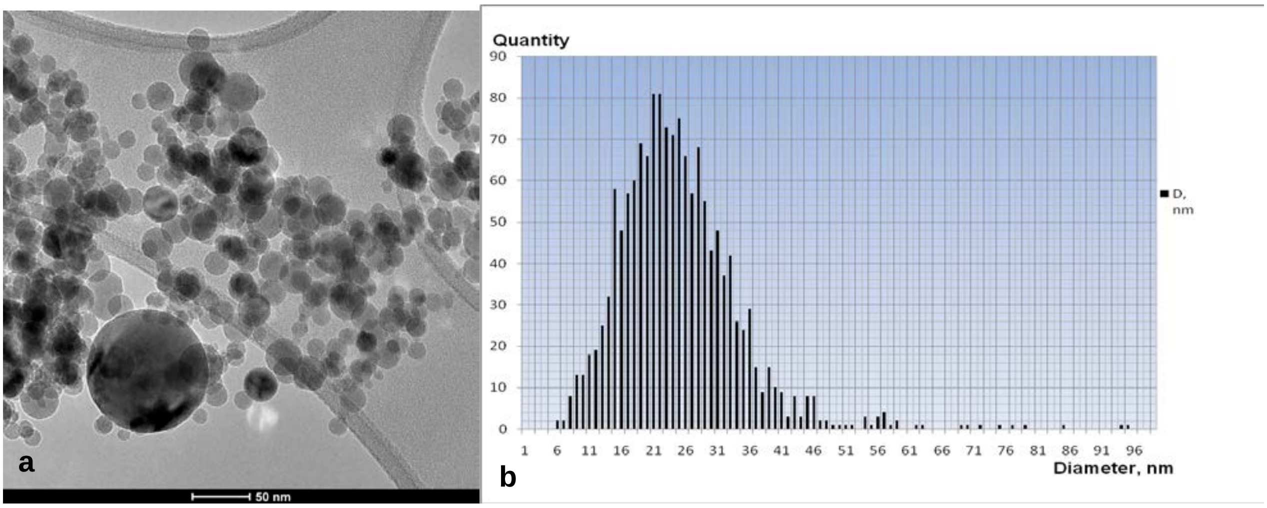

Figure 1. *Al_2O_3 nanoparticles at low magnification (120 kV), only one particle of large diameter (100 nm) is observed (a). The diameter distribution of small nanoparticles (b).*

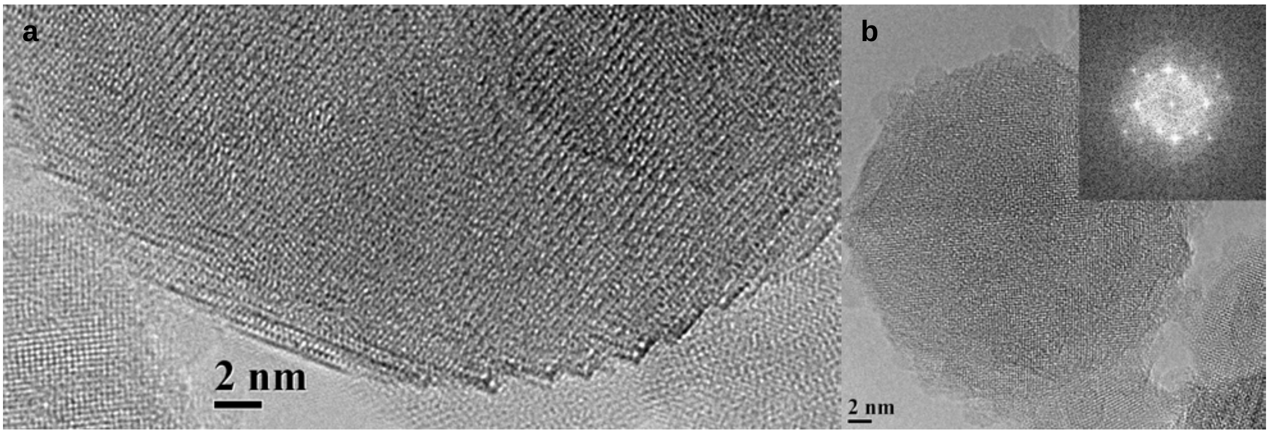

Figure 2. *HRTEM images of Al_2O_3 nanoparticles (300 kV). Atomic steps associated with growing process are visible (a). The BF image and its Fast Fourier transform of single cubic phase particle (b).*

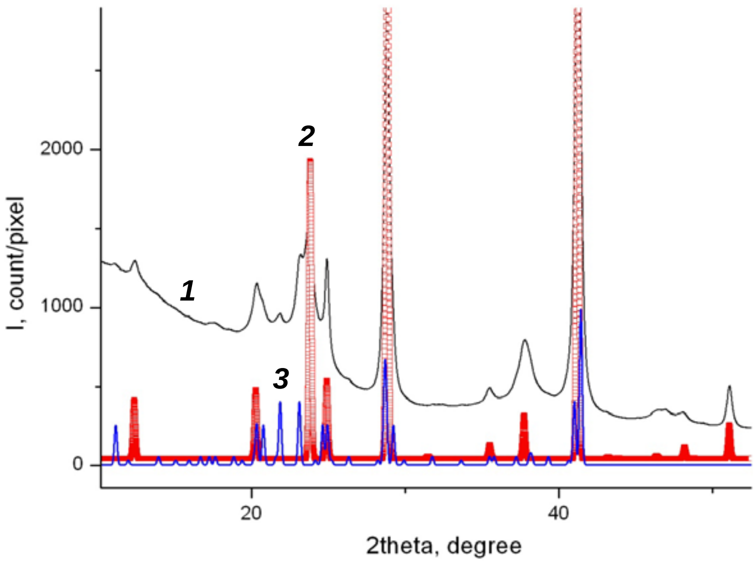

Figure 3. *Measured and calculated X-ray patterns: 1 – measured, 2 – calculated for cubic phase, 3 - calculated for orthorhombic phase.*