Nanocrystalline pure or doped ceria is an important material widely used in various fields of technology, including optics, microelectronics and catalysis. Doping of ceria with transition metal ions enhances its property and improves the thermal stability of nanocrystalline ceria against sintering. It has been established that catalytic activity of ceria nanoparticles depends strongly on their morphology: nanoparticles with cube or rod morphology, exposing {1 0 0} planes at the surface, are desirable for catalytic reactions of CO and soot combustion [1,2].

In this work mixed Ce1−xLn2−y (Ln=Gd, Er) oxides were synthesized by the hydrothermal treatment [3,4]. Two modifications of the hydrothermal treatment – classical and microwave assisted – were applied. The effect of the amount of dopant and the synthesis method on the phase composition and morphology of the of lanthanide oxides was studied by SEM-EDS, EBSD, TEM, XRD and Raman spectroscopy.

By classical hydrothermal treatment, for low doping level, nanocubes of the mixed Ce-Ln oxide with fluorite structure and bimodal size distribution (small 5-20 nm and much bigger 50-80 nm) were formed (Fig.1), while at higher doping (x > 0.3 ) rod-like particles of Ln hydroxide were also observed. Using of microwave radiation enabled the synthesis of the nanocubes of the mixed oxides at significantly shorter time, but the resulting materials is different: over broad range of Ln contents (0.05 <x< 0.5) particles with nanorod and nanocube morphology were obtained (Fig.2). TEM show that smallest particles with low doping level, which could not be characterized by SEM, contains mostly regular cube shape particles, though there is a fraction of small particles having rounded corners (Fig.3). SAED pattern contain sharp rings that can be assigned to fluorite structure of ceria. Particle size distribution is very broad and bimodal.

EBSD combined with EDS was used to analyze the structure and composition of unusual, large oxide nanocubes (50 – 80 nm) appearing in the samples (Fig.4). It appeared that the nanocubes of the mixed Ce-Ln oxide have fluorite type structure of CeO2 and are single crystals but not aggregates of smaller crystallites.

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Fig. 1: SEM image from $\text{Ce}_{0.95}\text{Er}_{0.05}\text{O}_{2-y}$ (classical hydrothermal treatment)

Fig. 2: SEM image from $\text{Ce}_{0.95}\text{Er}_{0.05}\text{O}_{2-y}$ (microwave assisted hydrothermal treatment)

Fig. 3: TEM image and SAED pattern from $\text{Ce}_{0.95}\text{Er}_{0.05}\text{O}_{2-y}$ (classical hydrothermal treatment)

Fig. 4: EBSD indexed pattern from single nanocube of $\text{Ce}_{0.95}\text{Er}_{0.05}\text{O}_{2-y}$