FIFTEENTH YOUNG RESEARCHERS' CONFERENCE MATERIALS SCIENCE AND ENGINEERING

December 7-9, 2016, Belgrade, Serbia Serbian Academy of Sciences and Arts, Knez Mihailova 36

PROGRAMME & THE BOOK OF ABSTRACTS

MATERIALS RESEARCH SOCIETY OF SERBIA INSTITUTE OF TECHNICAL SCIENCES OF SASA

December 2016, Belgrade, Serbia

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## Investigation of changes in positronium trapping in pores under the water influence in nanostructured MgO- Al<sub>2</sub>O<sub>3</sub> ceramics

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It is known that nanostructured MgO-Al<sub>2</sub>O<sub>3</sub> ceramics are one of the best materials for humidity sensors. The functionality of such ceramics is dependent on microstructure of grains, grain boundaries and pores. These elements significantly affect on their nanostructurization. In addition, electrical properties of ceramics depend on the sorption processes in these materials. In this work we used positron annihilation lifetime (PAL) spectroscopy to investigation of changes in PAL parameters in nanopores MgO-Al<sub>2</sub>O<sub>3</sub> ceramics sintered at 1100  $^{\circ}$ C at different stage of water-immersion and drying. The PAL investigation were performed using ORTEC spectrometer (22Na source) in the ceramic samples dried in vacuum at 120  $^{\circ}$ C for 4 hours as well as after 1, 2, 3 and 7 days immersion in water vapor. The four-component fitting procedure with positron lifetimes tau1, tau2, tau3 and tau4 and intensities I1, I2, I3 and I4 was used for analysis of PAL spectra.

It is established that the main changes caused by sorption of water are observed in the third and fourth positronium (o-Ps-related) components. The number of nanopores corresponds to the intensity of these components. Thus, the intensity I4, which corresponds to the number of larger nanopore with radius of ~ 18 Å, significantly decreases after 1 day after water-immersion. The lifetime tau4 decreases, which reflects lessening of nanopore radius calculated within Tao-Eldrup model. It is connected with penetration of nanopores by water molecules (fully or partially filling) changing characteristics of o-Ps-related component. Obviously, such pores should have access to environmental and internal communications at the nanoscale. Additional studies of MgO-Al<sub>2</sub>O<sub>3</sub> ceramics after 2, 3 and 7 days after water-immersion shows a gradual increase in the lifetime tau4 and intensity I4, indicating release of water from the inner voids of ceramics. After final drying in vacuum at 120 °C for 4 hours, the initial distribution of pores in MgO-Al<sub>2</sub>O<sub>3</sub> ceramics tends to recovery. However, the parameters of the fourth component are not fully recovered, testified that some water molecules remaining adsorbed.

The most significant changes caused by water sorption processes are observed for the largest nanopore. The intensity of this component does not return to initial value after drying of ceramics, because not all water released into the nanopore interior. Reducing the value of the lifetime tau4 after drying of ceramics with poorly developed porosity can be due to the formation of thin layers of water molecules surrounding the large pores that completely freed moisture at 120 °C. The lifetime tau3 decreases after water-immersion of ceramics with a gradual increase in drying and intensity I3 grows, indicating annihilation of o-Ps in water-filled nanopores. Water in the nanopores of smaller radius ~3 Å after drying, reflects in increasing of intensity I3 and a slight decreasing of lifetime t3. It is noted that the lifetime of

 $\sim$  1,8 nm reflects the annihilation in the water "bubbles" with radius near 3 Å. Its number increases in accordance with intensity I3.

Thus, it is shown that lifetimes of third and fourth positronium (o-Ps)-related component of PAL spectra decreases in water-immersed MgO-Al<sub>2</sub>O<sub>3</sub> ceramics reflected decreasing of free-volume after water-immersion. The amount of biggest nanopores decreases, while positronium trapping in smaller nanopores carried out simultaneously with annihilation in water "bubbles".

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## Ethylenediaminetetraacetic acid (EDTA) assisted hydro/solvothermal synthesis of up-converting rare earth fluorides

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Over the last decade, a lot of effort was directed toward developing of the representative methodologies for the preparation of up-converting (UC) particles which exhibit a unique narrow visible emission when excited by lower-energy photon radiation. This work presents the impact of different processing parameters on structural, morphological and optical properties of up-converting (UC) rare earth fluorides obtained by hydro/solvothermal synthesis. Monodisperse NaYF<sub>4</sub>:Yb<sub>3</sub>,  $Er_{3+}$  particles with different morphology, size and crystal phase were synthesized with a help of ethylenediaminetetraacetic acid (EDTA) through adjusting the precursor concentration, degree of doping, polarity of solvent and reaction time. They are characterized by X-ray powder diffraction, scanning and transmission electron microscopy, energy dispersive X-ray and Furrier transform infrared spectroscopy, as well as photoluminescence measurements. It was shown that particle size and phase composition are dependent on the precursor concentration, type of solvent and doping degree, while the cubic to hexagonal transformation of NaYF<sub>4</sub>:Yb<sub>3+</sub>/Er<sub>3+</sub> phase is affected by the reaction time. The crystallization of the orthorhombic  $YF_3$ :  $Yb_{3+}/Er_{3+}$  phase is established either after decreasing concentration of dopants or increasing polarity of solvents. All of the synthesized particles exhibited efficient up-conversion emission which can be tuned from pure green to the yellowish-orange through control of particles size and phase composition.