Streszczenie

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Combined positron-annihilation and structural studies of hydrothermally grown zirconia

Zirconia (ZrO_2) finds numerous applications from jewellery to dentistic ceramics but is usually stabilized by yttrium. In hydrothermally microwave grown zirconia the stechiometric ratio of O to Zr can vary and the material shows extended nano-porosity. Therefore it creates an opportunity for proper applications as high-temperature sensor of oxygen in exhaust gases, optical oxygen sensor and as high temperature membrane in fuel cells. Complementary positron and HREM techniques allow to study the porosity, presence of defects in the crystalline structure and the chemical surroundings of defects.
Samples were prepared in Warsaw laboratory from zirconia nanopowders obtained in hydrothermal microwave driven process followed by annealing up to 900°C, and then annealed under variable oxygen pressure. Positron measurements have been performed with two techniques: 1) Doppler broadening in Trento laboratory, using an electrostatic slow positron beam tunable in the 0.05-25 keV energy range allowing to scan to a depth scale from 1 to about 1000 nm; 2) lifetime technique in Toruń laboratory with 180 ps time resolution performing measurements in the material bulk. In beam measurements a high fraction (8-11%) of ortho-positronium has been detected, indicating a nano-porous structure within the whole material but particularly down to first 20 nm. The porosity lowers after annealing in oxygen atmosphere is lower in samples annealed at 800ºC than in samples annealed at 700ºC; the difference in annealing temperature does not influence the porosity of surface layers. Also from bulk lifetime measurements we deduce on presence of nano-pores in all samples annealed in the 700-900ºC temperature range – apart from short (180 ps) lifetime component typical for the ZrO_2 crystalline phase we note a longer component (350-370 ps). This fraction of that second component reaches a maximum at 800ºC (70%) compared to only 20% at 700ºC annealing temperature. Also High-Resolution Electron Microscopy structural studies for ZrO_2 nanopowder annealed at 800°C carried out at St. Andrews confirmed the presence of different defects such as stacking faults, voids within ~ 2-4 interplanar distance and terraces. This is a clear hint for further, functional studies of this material.

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