Supplementary Material (ESI) for Journal of Materials Chemistry

Electronic Supplementary Information

Single-crystal nanofibers of Zr-doped new structured $PbTiO_3$: hydrothermal synthesis, characterization and phase transformation

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Materials and methods

Chemistry grade tetrabutyl titanate ($(C_4H_9O)_4Ti$), zirconium (IV) n-butoxide ($(C_4H_9O)_4Zr$), and lead nitrate ($Pb(NO_3)_2$) were used as the starting materials. Potassium hydroxide (KOH) and 2g/l poly (vinyl alcohol) (PVA) were used as mineralizer and surfactant, respectively. The Zr-doped new structured $PbTiO_3$ samples were synthesized via a hydrothermal method as follows: The atomic ratio of Pb / (Ti+Zr) was set at 1 and the concentration of Zr doping was adjusted as the atomic ratio of Zr / (Ti+Zr) (0, 5%, 10%, 15% and 20%). Firstly, the appropriate quantity of $(C_4H_9O)_4Ti$ and $(C_4H_9O)_4Zr$ were dissolved in 2-methoxyethanol to form the solution. Subsequently, approximately 2ml ammonia was added to obtain the white coprecipitation in the solution. In order to remove the ammonium ions, the precipitation was filtered with deionized water for three times. Subsequently, the precipitation was mixed with $Pb(NO_3)_2$ aqueous solution, KOH solution and 10ml PVA. After stirring for 1h, the resulting precursor suspension was moved into the 50ml stainless-steel Teflon-lined autoclave for the hydrothermal treatment. The autoclave was sealed and held at 200 ^oC for 12, 11, 10, 8.5 and 6 hours for the solutions with different Zr / (Ti+Zr) ratio, respectively. Finally, it was cooled down to room temperature naturally. The resultant products were filtered and washed with deionized water and ethanol, and subsequently

air-dried at 80 ^{o}C for the characterization. All the powders were heat treated at 650 ^{o}C in air for 60 to 100 minutes for the phase transition.

Characterization

X-ray power diffraction patterns were collected at room temperature on a Thermo ARL X'TRA power diffractometer with Bragg-Brentano geometry by Cu Kα radiation (λ = 1.54056 Å) under 0.82 Å resolution. The microstructure characteristics were studied using a field emission scanning electron microscope (Hitachi MODEL S-4800) equipped with an EDS detector. The elemental composition and quantitative analysis on nanofibers were performed by scanning a large area of nanofibers with EDS for six times, and these results were used to calculate the average values and the standard deviations. Transmission electron microscope (TEM) images, HRTEM images, and SEAD patterns were taken via a transmission electron microscope (JEOL-2010) using an accelerating voltage of 200kV (with a double-tilt holder).