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Short Communication

$Pb(Zr_{0.52}Ti_{0.48})O_3$ nanotubes synthesis and infrared absorption properties

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ABSTRACT

Herein a useful methodology to synthesize the lead zirconate titanate (PZT) nanotubes via a dip-coating deposition process with anodic aluminum oxide (AAO) template is proposed. The nano-porous AAO templates were produced using a controlled two-step electrochemical anodization technique. The PZT/AAO composite was formed using the dip-coating wetting technique. The prepared PZT precursor solution was driven into the nanopore channels of AAO template under the driving force of capillary action, subsequently the sintering process of the as-filled templates was carefully tuned to obtain $Pb(Zr_{0.52}Ti_{0.48})O_3$ nanotubes of crystalline tetragonal phase with uniform pore size and ordered arrange. Fourier transform infrared spectroscopy (FTIR) results show that in the 1200–1900 cm^{-1} band, the composite structure of PZT/AAO has obvious absorption peaks at 1471.56 cm^{-1} and 1556.09 cm^{-1}, the absorption intensity of the composite structure is about six times of pure AAO template. The unusual optical properties found in PZT/AAO composite will stimulate further theoretical and experimental interests in ferroelectric nanostructures.

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1. Introduction

In recent years, investigation of the pyroelectric effect in ferroelectric nano materials for uncooled infrared detectors has become of great interest. The nanoscale ferroelectric infrared detectors are expected to yield better sensitivity and faster response than equivalent ceramic and bulk single crystals [1,2]. As one of the most widely used ferroelectric materials with high spontaneous polarization, dielectric permittivity and piezoelectric coefficients, PZT is widely used as the pyroelectric element in the IR radiation detection application [3,4]. Many different types of uncooled pyroelectric PZT sensors with various nanostructure have been designed and developed to improve their device performance, including PZT nanoislands [5], PZT thin film [6], PZT nanoparticles etc [7]. However, up to date, very few reports about infrared absorption properties of PZT nanotubes are published despite relevance of PZT nanotubes, which can improve the performance of PZT and their device applications which do not exist in the individual bulk materials [8–14]. From both fundamental and technological points of view, any theoretical studies will be useful in terms of PZT nanotubes.

In this article, we report that $Pb(Zr_{0.52}Ti_{0.48})O_3$ nanotubes with uniform pore size and ordered arrange were successfully synthesized. The sol layer, containing lead, zirconium and titanium precursors, formed on the pore walls of AAO template. Dip-coating deposition was utilized to grow PZT nanotubes through AAO template which offered preferable tailoring of the dimensions of the nanopores. The morphology of PZT nanotubes were examined by scanning electron microscopy (SEM). The X-ray diffraction analyses indicated the presence of tetragonal phase, and energy dispersive X-ray (EDS) elemental analyses revealed the presence of constituting elements of PZT. The infrared absorption of PZT/AAO composite structure at 1200–1900 cm^{-1} band was discussed.

2. Experimental procedure

2.1. Template production

The AAO template was fabricated through the well-known two-step anodization process as with our previous work [15,16]. In brief, annealing, organic cleaning and electrochemical polishing were taken on the 0.2-mm-thick aluminum (Al) foil (99.999% in purity) prior to the anodization process, to increase the grain size and reduce the surface roughness, leading to a better homogeneity for the development of the nanopores. The first anodization was

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carried out in oxalic acid solution for 4 h at the temperature of 5 °C and voltage of 40 V (best arrangement in oxalic acid) [17], followed by the 6 wt% H_3PO_4 and 1.8 wt% H_2CrO_4 aqueous solution to remove the oxide layer. The second anodization lasted for 5 h with the same anodizing parameters, and the pore-widening process was carried out for 150 s in 5 wt% H_3PO_4 solution at 60 °C. Then, the aluminum substrate was dissolved in a saturated aqueous solution of $CuCl_2$ dissolved in water before through-hole AAO was realized in 5 wt% H_3PO_4 solution for 8 min at 50 °C.

2.2. Sol preparation and deposition

A conventional sol–gel process was used to synthesize PZT in the desired chemical composition of $Pb(Zr_{0.52}Ti_{0.48})O_3$. Lead acetate trihydrate (99.5%), Zirconium(Ⅳ) nitrate (70.0%) and tetrabutyl titanate (99.0%) were used to prepare the PZT precursor solution

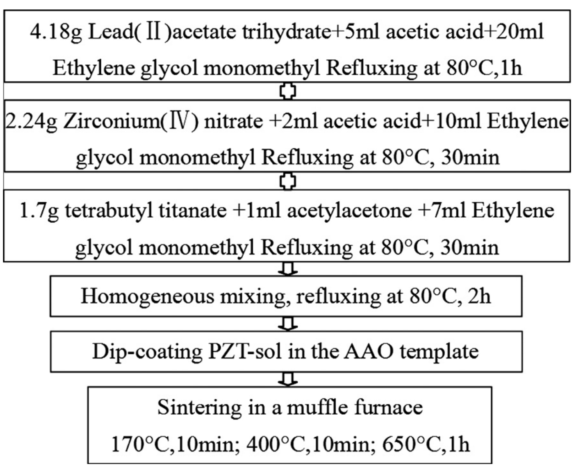

Fig. 1. Flow diagram for the synthesis of the PZT nanotubes.

having cationic (Pb/Zr/Ti) ratios of 1.1/0.52/0.48. Excess lead acetate (10 mol%) was added to compensate Pb-loss during thermal processing [18]. The solution route was similar to the methodology of Yi and Sayer, as reported elsewhere [19,20]. Fig. 1 shows flow diagram for the synthesis of the $Pb(Zr_{0.52}Ti_{0.48})O_3$ nanotubes. As-prepared sol was dropped on the surface of an AAO template to dip for 20 min, followed by spin coating with a rotation speed of 3000 rpm for 60 s, and then as-prepared PZT/AAO was dried at 120 °C on hot plate for 60 s. This process was repeated to obtain the desired PZT nanotube wall thickness. Sequentially it was sintered under ambient conditions. The PZT nanotubes was released from AAO template by being dissolved in 2 M/L NaOH solution.

2.3. Characterization

The morphologies of as-prepared samples were characterized by Nova Nano-SEM 450 scanning electron microscopy in Fig. 2. The phase assemblage of specimens was examined by X-ray diffraction (XRD) (D8, Brucker) using Cu Ka (1.5406 Å) radiation and a graphite monochromator in 2 h range of 10–80° in Fig. 3. EDS mapping of SEM reveals the presence of all elements of PZT in Fig. 4. FTIR absorption measurements were performed using a Fourier transform infrared spectrometer (Bruker VERTEX 70) in Fig. 5.

3. Results and discussion

SEM was employed to investigate the morphologies of the grown structures. Fig. 2a is the SEM planar view image of the as-prepared AAO with uniform and ordered array. The template channels in Fig. 2b clearly contain the PZT nanotubes. To investigate the free PZT nanotubes, the alumina membrane was chemically partial etched away for 5 min in Fig. 2c or fully etched away for 1 h in Fig. 2d using 2 M/L NaOH solution. It can be seen that the template contains the arrays of hollow and long PZT nanotubes, rather than nanowires or rods from Fig. 2b and c, which indicate the nanotubes

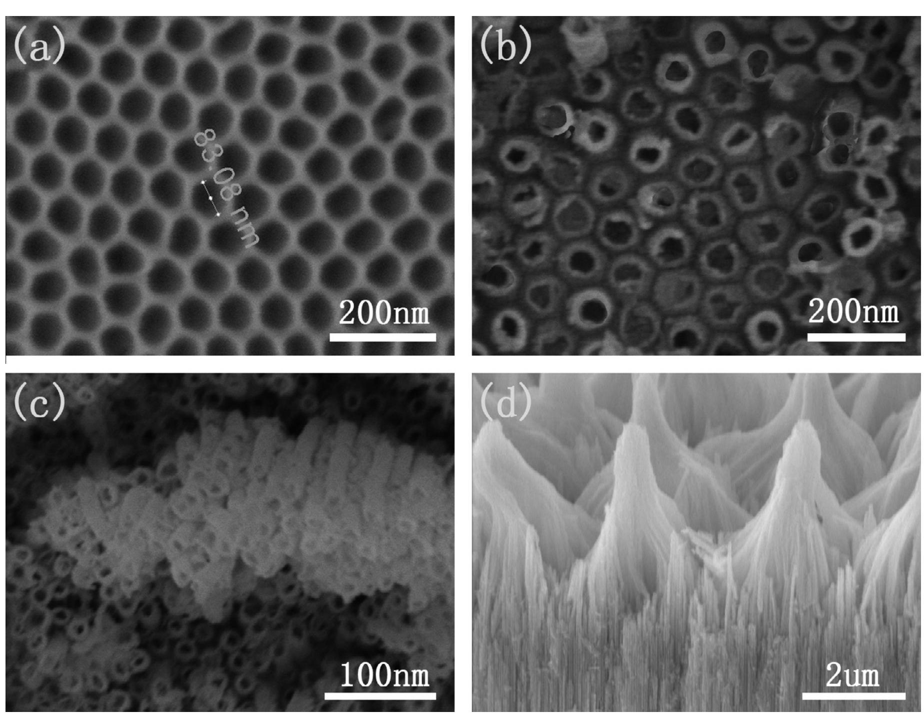

Fig. 2. SEM images of (a) an AAO template, (b) an AAO template filled with PZT nanotubes, (c) free PZT nanotubes, and (d) clustered PZT nanotubes without scaffold of AAO.

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