



Quantum confinement of lead titanate nanocrystals by wet chemical method



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ABSTRACT

Lead Titanate (PbTiO₃) is a category of the practical semiconductor metal oxides, which is widely applied in various scientific and industrial fields because of its catalytic, optical, and electrical properties. PbTiO₃ nanocrystalline materials have attracted a wide attention due to their unique properties. PbTiO₃ nanocrystals were investigated by X-ray diffraction (XRD) to identify the PbTiO₃ nanocrystals were composed a tetragonal structure. The diameter of a single sphere was around 20 nm and the diameter reached up to 3 μm. The chemical composition of the samples and the valence states of elements were determined by X-ray photoelectron spectroscopy (XPS) in detail.

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1. Introduction

Recently, one-dimensional (1D) nanostructures have stimulated much attention because of their fascinating applications for well-defined interconnects and building blocks for nanodevices. Nanocrystalline materials have attracted a wide attention due to their unique properties and immense potential application for opto-device fabrication of one-dimensional (1D) nanostructures, such as nanotubes, nanobelts, nanowires and nanorods because of the distinct geometries, and novel physical and chemical properties of different from those of bulk counterparts [1–3]. Due to the size confinement in the radial direction materials of these nanostructures are promising candidates for realizing nanoscale electronic [4,5] optical and magnetic [6–10] devices. Ferroelectric oxides represents a particular interesting class of materials, which exhibits spontaneous polarization that can be reoriented by

external electric field, and possess a broad range of properties, such as remnant polarization and high dielectric permittivity as well as piezoelectricity and pyroelectricity [11,12]. Previous studies on thin film and nanocrystalline samples have indicated that their physical properties are critically dependent on their dimensions [13]. Thus, it is of great significant to investigate the fabrication and physical properties of ferroelectric of 1D nanostructure. So far, long ferroelectric nanowires with well-defined structures and a diameter of 5 nm–10 nm have been fabricated by using different methods [14,15]. For example, Urban and co-workers have fabricated well-isolated nanowires of BaTiO₃ and SrTiO₃ with diameters ranging from 10 to 50 nm and lengths reaching up to 10 μm by solution-phase decomposition of bimetallic alkoxide precursors in the presence of coordinating ligands [16]. Deng et al. have fabricated single crystalline PbTiO₃ nanorods (NRs) with diameters of 50 nm–80 nm by solid-state reaction [17]. Hydrothermal technique is a promising way for fabricating ferroelectric nanomaterials because it can realize a low processing temperature of 200 °C or less, and can obtain products with high purity [18]. For example, Xu et al. have fabricated single-crystalline tetragonal perovskite NRs and nanowires (NWs) using hydrothermal process assisted by polymers [19]. As a ferroelectric material, PbTiO₃ exhibits a

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perovskite structure and a high Curie temperature ($T_C = 763$ K) compared to other ferroelectric materials, such as BaTiO_3 , SrTiO_3 etc., which makes it useful over a wide temperature range. It has many potential applications in electronic and microelectronic devices, belonging to the most important ferroelectric and piezoelectric families [20–22]. For this material, many early studies were mainly concerned with thin films and particles [23]. Although high quality nanosphere were thought to be quite difficult to obtain. Therefore, there is considerable interest for studying such materials not only for future applications but also from a fundamental point of view.

2. Experimental

2.1. Chemicals & typical synthetic process of lead titanate nanocrystal

All chemical reagents (analytical grade) were used as received without further purification. Lead titanate (PbTiO_3) were used as the starting materials, and Di-methyl formaldehyde (DMF) was served as a capping agent. In 1 mol % of PbTiO_3 was dissolved in 100 ml deionized water and 1 mol % of DMF was dissolved in 50 ml ethanol solution. After ultrasonication for an hour the suspension was transferred into 150 ml Teflon lined stainless steel autoclave. The autoclave was maintained at a temperature of 390°C for 12 h. After cooling to room temperature (RT) naturally, the black yellowish product was washed with distilled water for several times and then dried under vacuum at 110°C for 36 h. Then the black yellowish dispersion was rinsed three times with deionized water by centrifugation. Finally nearly 1 g of H_2SO_4 was added into the suspension and stirred for 5 h. The as-obtained samples were filtered and washed 3 times with distilled water and ethanol and then dried in vacuum at 90°C for 7 h.

2.2. Sample characterization

The X-ray powder diffraction (XRD) experiments were measured on a Rigaku D/max-RB diffractometer with Ni-filtered graphite monochromatized $\text{CuK}\alpha$ radiation ($\lambda = 1.54056 \text{ \AA}$) under 40 kV, 30 mA and scanning between 10° and 90° (2θ). The XPS spectrum was recorded on a ESCALAB 250 photoelectron spectrometer (Thermo-VG Scientific, USA) with Al $\text{K}\alpha$ (1486.6 eV) as the X-ray source. High-resolution Transmission electron microscopy (HRTEM) measurements were made on a HITACHI H-8100 electron microscopy (Hitachi, Tokyo, Japan) with an accelerating voltage of 200 kV. The sample for HRTEM characterization was prepared by placing a drop of colloidal solution on carbon-coated copper grid and dried at room temperature. The elemental composition was determined using the selected area electron diffraction (SAED) (IH-300X) analysis was performed at several points in the HRTEM system respectively.

3. Results and discussion

3.1. X-ray powder diffraction

Fig. 1 shows the X-ray diffraction (XRD) pattern of PbTiO_3 nanocrystals sample by wet – chemical synthesized at 390°C for 12 h. Most of the diffraction peaks in this pattern can be assigned to a tetragonal phase with the lattice parameter, $a = 3.904 \text{ \AA}$, corresponding well with the reported data (JCPDS card file no. 01-074-2495). The strong and sharp reflection peaks suggest that the as-prepared products are well crystallized [24,25].

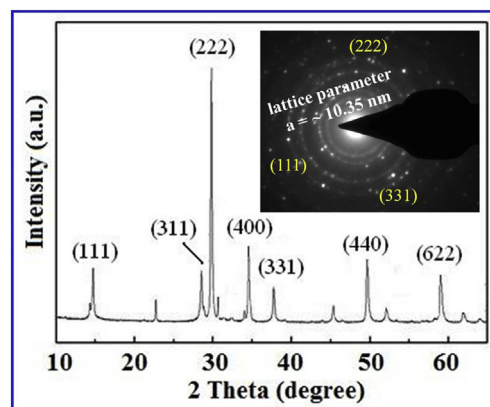


Fig. 1. X-ray powder diffractogram of PbTiO_3 nanosphere.

3.2. XPS analysis

Fig. 2 shows the XPS wide scan spectra of PbTiO_3 nanocrystals in the binding energy ranging up to 600 eV. It can be seen that the nanocrystals contains Pb, Ti and O elements and no other elements are detected expect for carbon, the atomic ratio of Pb:Ti:O is respectively. In two peaks at 325 eV and 545 eV for O1s were identified; each component peak in the spectrum was fitted with Lorentzian function respectively [26]. The major peak of lower binding energy was assigned to lattice oxygen (Ti–O–) in the PbTiO_3 nanocrystal, while the smaller peak of higher binding energy was assigned to the hydroxyl group of oxygen, which is very common in samples with high surface energy [27]. This may be due to the variation of the lattice constants a & c ($a = 0.391 \text{ \AA}$, $c = 0.415 \text{ \AA}$, which are calculated from XRD pattern), together with the shrinking of lattice for PbTiO_3 nanocrystal, which means that the oxide anions form octahedral of TiO_6 enclosing the titanium ions and the Ti–O bonds in PbTiO_3 lattice become much more stronger. This result reveals that the lattice shrinkage plays a significant role in the spin–orbit splitting of $\text{Ti}2p$ state.

3.3. High resolution scanning electron microscopy

The high-resolution transmission electron microscopy (HRTEM) of PbTiO_3 nanocrystals prepared by wet-chemical route. It is obvious from Fig. 1 that the XRD and SAED patterns are densely

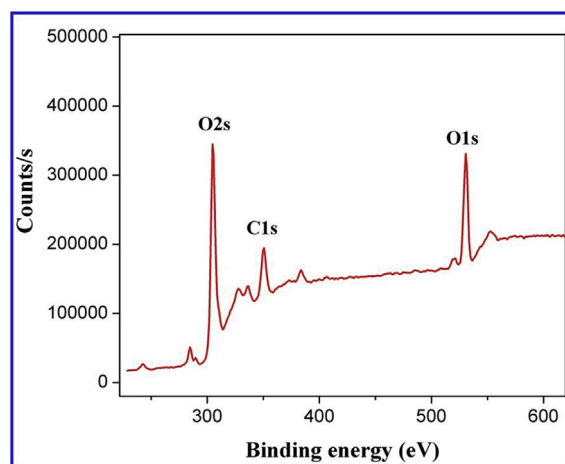


Fig. 2. XPS wide scan spectra of PbTiO_3 nanosphere.

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