



Nanoembossing and piezoelectricity of ferroelectric $\text{Pb}(\text{Zr}_{0.3}\text{Ti}_{0.7})\text{O}_3$ nanowire arrays

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ABSTRACT

Arrays of ferroelectric PZT nanowires with lateral size down to 200 nm were fabricated by nanoembossing technology. Structural characterization of the embossed PZT film was studied by Raman spectroscopy. Multidomain configurations of a single nanowire have been explored by vertical mode piezoresponse force microscopy (VPFM). The local electric polarization of the individual ferroelectric nanowire has also been investigated. Excellent ferroelectric and piezoelectric characteristics observed in the embossed PZT nanowires suggest nanoembossing technique proposed in this work is promising to become a useful method for ferroelectric nanowires fabrication.

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1. Introduction

The broad potential applications in nanoscale piezoelectric transducers, ultrasonic devices, and non-volatile memory devices [1–4] have been leading to increasing efforts in fabrication and understanding of ferroelectric nanostructures that could engineer domain architectures at nanoscale and induce intriguing ferroelectric and piezoelectric characteristics [5–7]. Fabrications of ferroelectric nanowires have been reported using different methods including infiltration [8], molten salt technique [9], electrophoresis [10], solvothermal [11]. Most of the approaches are based on a chemical process and normally time consuming. The conventional photo lithography [12,13] to pattern submicron and nanoscale structures has been reported damages and defects introduced in ferroelectrics, which can significantly degrade ferro/piezoelectric properties. Better way to control the quality of ferroelectric nanostructures and alternative patterning techniques are thus highly demanded. Nanoembossing has emerged as a fast, convenient and high-resolution patterning technology for fabricating nanostructures in which surface patterns of a template are replicated into a material by mechanical contact and

three-dimensional material displacements [14,15]. In this work, we have applied this technique on ferroelectric lead zirconate titanate [$\text{Pb}(\text{Zr}_{0.3}\text{Ti}_{0.7})\text{O}_3$ (PZT)] to form nanowires with strong piezoelectric property. The PZT nanowires with width of ~200 nm and length of several microns were measured by scanning electron microscope (SEM) and atomic force microscope (AFM). The piezoelectric characteristics of an individual PZT nanowire was demonstrated by piezoresponse force microscopy (PFM). The PFM technique is based on the detection of local piezoelectric deformation of a ferroelectric sample induced by an external electric field. The amplitude of the vibration signal accounts for the magnitude of the piezoelectric coefficient of the sample, while the phase signal illuminates the domain polarity direction, details are reported elsewhere [16–18]. Raman spectroscopy was performed to elucidate the ferroelectric phase of the embossed PZT film.

2. Experimental details

Ferroelectric thin film PZT was prepared on Pt/Ti/SiO₂/Si substrate by a sol–gel method with a ratio of 30:70, in which composition PZT films exhibited less anisotropy in remnant polarization along different crystalline textures [19]. The starting materials were lead acetate trihydrate [$\text{Pb}(\text{OCOCH}_3)_2 \cdot 3\text{H}_2\text{O}$, 99.5%], zirconium tetra *n*-propoxide ($\text{Zr}(\text{OC}_3\text{H}_7)_4$, 70%), titanium (IV) butoxide ($\text{Ti}(\text{OC}_4\text{H}_9)_4$, 98%) as a precursor material and methanol/acetic acid mixed solvent as a solvent [20]. Fig. 1a shows the nanoembossing process of ferroelectric PZT. After spin-on, the precursor film was first baked on a hotplate at 70 °C in air atmosphere for 5 min. The pre-bake process played two important roles here, one was the

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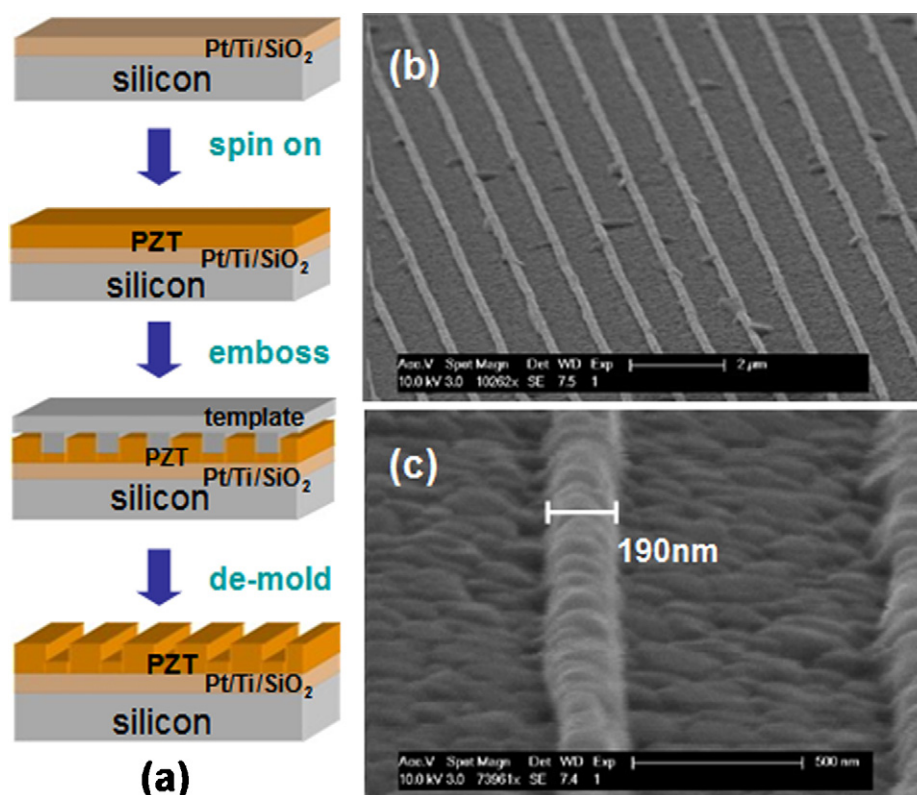


Fig. 1. (a) Schematic illustration of nanoembossing process of ferroelectric PZT nanowires. (b) PZT nanowire arrays with several microns in length measured by SEM. (c) A single PZT nanowire with width of 190 nm.

evaporation of the solvent in the coated film, and the other was the moderation of the precursor's viscosity [21]. An anti-sticking layer was coated on a silicon template surface prior in order to reduce its adhesion to the PZT gel. Then a nanoembossing process was carried out under a pressure of ~ 10 MPa for 15 min using the pretreated silicon template with 750 nm line widths and 1 μm in period. After embossing, the gel layers were first pyrolyzed in air on a hotplate at 350 $^{\circ}\text{C}$ for 5 min and then crystallized by conventional thermal annealing in air at 650 $^{\circ}\text{C}$ for 15 min.

Raman measurement was performed using a RENISHAW inVia Micro-Raman System equipped with a Peltier cooled CCD camera and a Leica microscope. The experiment was carried out at an excitation wavelength of 514.5 nm from an Ar^+ laser (20 mW) by focusing the laser beam into a ~ 1 μm -diameter spot on ferroelectric samples at room temperature. The scattered light was detected in the backscattering geometry [21].

PFM characterizations of embossed PZT films were employed using an AFM (Veeco Multimode V) operated in contact mode at room temperature. The piezoresponse signals were measured using a lock-in amplifier and a Pt-coated AFM tip with a force constant of 0.03–0.2 N/m, resonant frequency of 14–28 kHz. An AC voltage of 1.2 V at frequency of 100–200 kHz was applied to measure the in-field nanoscale hysteresis loops.

3. Results and discussion

Fig. 1b shows the embossed highly ordered PZT nanowire arrays with length of several microns measured by SEM. One can see in Fig. 1c that the PZT single nanowire has a width of about 190 nm, in which PZT grains assembled orderly. Raman scattering spectrum of an embossed PZT region is shown in Fig. 2, which matched well with typical Raman peaks of distorted perovskite PZT [22]. The prominently intense, low-frequency modes at 140.6 and 195.4 cm^{-1} corresponded to $A_1(1\text{TO})$ and $E(2\text{TO})_T$ mode, respectively. The peak

at 502.9 and 599 cm^{-1} related to $E(3\text{TO})_T$ and $A_1(3\text{TO})$ mode. These four modes marked in Fig. 2 indicated that the embossed region had the tetragonal structure rather than the rhombohedral structures [23].

Fig. 3a shows the topography of a single embossed PZT nanowire measured by AFM. Its sectional profile (Fig. 3b) indicated the embossed depth was ~ 140 nm. The corresponding as-grown domain polarity state of the individual embossed nanowire was measured by vertical mode PFM (VPFM). As shown in Fig. 3c, there was a multidomain state in the single embossed nanowire, where the bright to dark contrast change represented domains with polarization vectors varying from upwards to downwards, respectively. From the sectional profile along the red line in Fig. 3c, we can

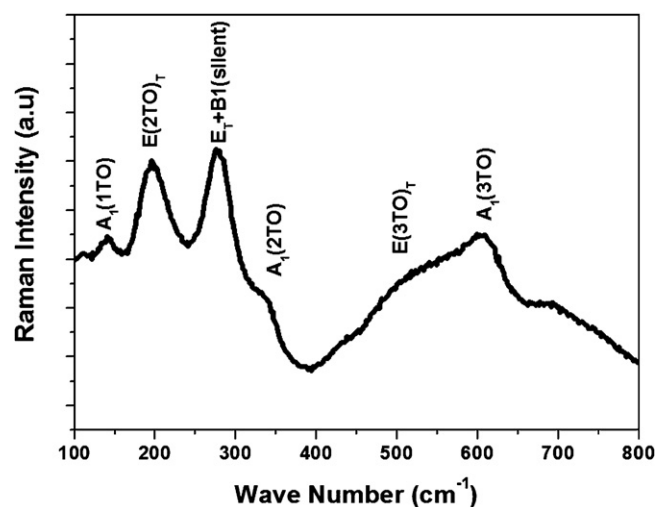


Fig. 2. Raman scattering spectrum of an embossed PZT region measured at room temperature.

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