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Water-Immersible Micromachined $Pb(Zr,Ti)O_3$ Thin Film Actuators

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Abstract. We demonstrate a water-immersible thin film lead zirconate titanate, $Pb(Zr,Ti)O_3$, [PZT] actuator, without special passivation layer, towards in-vivo or in-vitro scanning probe microscope (SPM) measurements of living cells in water or biological fluids. In order to be water-immersible, the electrodes need to be electrically insulated and the piezoelectric layer needs to be protected against direct water contact. This paper describes our design solution with a simple fabrication process for a water-immersible piezoelectric device, which separates the bottom electrode from the top electrode by having a narrow ditch covered with PZT film. The PZT film is then encapsulated with the top metal electrode without insulation layer. In this structure, the PZT is sandwiched between the top and bottom metal electrodes to prevent water permeation. The device is fabricated using lift-off processing for the bottom and top electrodes, sol-gel spinning for the PZT thin film and wet etching for the PZT patterning. The piezoelectric constant, d_{31}, is about -100 pC/N. The dielectric polarization and fatigue properties of the devices were measured in air and water. The spontaneous polarization, remnant polarization, coercive field and dielectric constant are 54 $\mu C/cm^2$, 15 $\mu C/cm^2$, 60 kV/cm and 1200, respectively. The polarization property of the device was unchanged in either air or water up to 1×10^9 continuous cycles.

Keywords: piezoelectric, actuator, thin film, water-immersible, MEMS

1. Introduction

The SPM technique has been a practical useful tool to observe and manipulate micro and nano-scale elements and phenomena. There have been many efforts to overcome two major problems in SPM technologies: slow scanning speed and inability to measure living cells underwater. Cantilevers with built-in thin film PZT actuators were developed by one of the authors and it was shown that the built-in PZT actuator cantilevers could make high speed imaging with AFM instruments [1]. PZT is an attractive material for microelectromechanical system (MEMS) actuators and sensors applications because PZT has many advantages such as ultra fine resolution, large force generation, fast expansion, no magnetic fields, low power consumption, vacuum and clean room compatibility and operation at cryogenic temperatures. There are many methods to fabricate the PZT films for micro-actuators, such as sol-gel spinning, sputtering, laser ablation, CVD, gas jet deposition or polished bulk PZT. Among the thin film fabrication methods, the sol-gel spinning process is convenient and cost effective, because the chemical composition of the PZT film is easily controlled and vacuum is not required.

In order to be water-immersible, the electrodes need to be electrically insulated as well as the piezoelectric layer needs to be protected against direct water contact. Passivation using an oxide layer on the cantilever surface to waterproof the PZT was previously reported [2]. However, the extra passivation layer on the cantilever degrades the performance of the cantilever actuator. In this paper, we demonstrate the water-immersible thin film PZT actuator without extra passivation layer. A new structure is designed for water-immersible piezoelectric actuation: the whole PZT is sandwiched between the top and bottom metal electrodes to prevent

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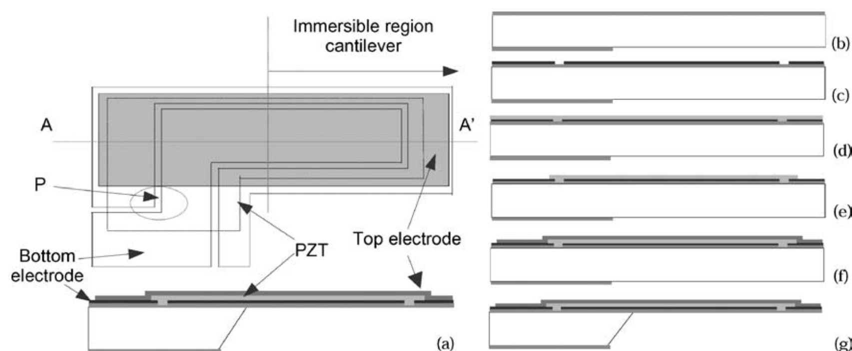
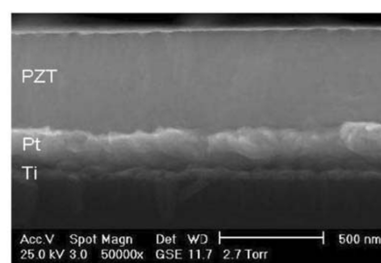


Fig. 1. Device schematic of the water-immersible piezoelectric actuator and the fabrication process flow: (a) Device schematic, (b) Thermal SiO_2 growth and creating a SiN_x KOH backside mask, (c) Pt/Ti bottom electrode lift-off, (d) PZT deposition and annealing, (e) PZT wet etch patterning, (f) Pt/Ti top electrode lift-off, and (g) Si backside KOH and RIE release etch.

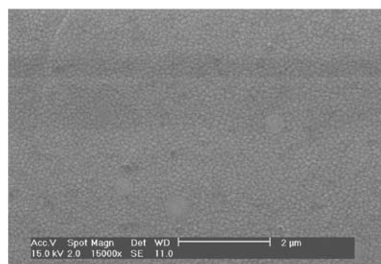
water permeation. The polarization property of the immersible PZT device in water has the same characteristics as in air up to 1×10^9 continuous cycles. In addition to SPM in liquid, this immersible PZT device can also be used for direct actuation of microfluidic devices in various wet applications.

2. Device Structure and Fabrication

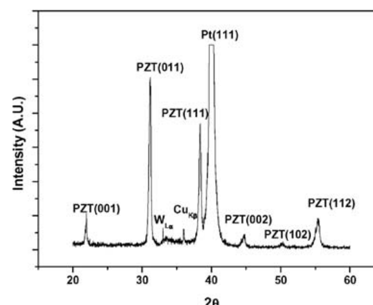
Figure 1(a) shows top and cross-sectional views of the designed immersible thin film piezoelectric cantilever. The width of the ditch on the bottom electrode is the key design parameter for both electrical separation, water insulation and good microstructure of PZT across the ditch. We tested several widths of the ditch, and achieved good dense PZT microstructure and electrical insulation with a 3 μm ditch width. The whole PZT film is then completely encapsulated by the top metal electrode without additional insulation layer, which forms the envelope together with the metal layer left outside the ditch of the lower electrode. Fabrication of the immersible piezoelectric thin film actuator involves both surface and bulk micromachining. Figures 1(b) to (g) show the fabrication process flow. First, 200 nm thermal oxide is grown on both sides of Si wafer. A 200 nm silicon nitride layer is deposited on backside via PECVD and then patterned to form a hard-mask for a KOH backside etching later. A 200/20 nm Pt/Ti layer is then evaporated on the substrate and patterned via lift-off. Lift-off permits the separated bottom electrode to be defined easily because the Pt film is not easy to etch using RIE or a wet etching process. The composition of the PZT solution (Mitsubishi Materials Corporation)



(a) Cross-sectional image



(b) Surface image



(c) XRD pattern

Fig. 2. SEM image and XRD pattern of fabricated PZT thin film on $Pt/Ti/SiO_2$. (a) Cross-sectional image, (b) Surface image and (c) XRD pattern.

is 52/48 as Zr/Ti ratio, and Pb content, which means Pb/(Zr+Ti), is 118/100. The solution is subsequently spun on to the substrate at 500 rpm for 3 secs and 3000 rpm for 30 secs. The precursor gel film is pyrolyzed at 350°C for 5 mins on a hot plate in several repeated steps to create a PZT layer with 0.5 um thickness. The PZT film is then annealed at 700°C for 15 mins. The piezoelectric and oxide membrane layers are patterned via wet-etching with an HCl: BOE (7:1): DI water (volume ratio of 100:16:200) etchant for 2 minutes [3, 4]. The PZT film can be etched before annealing at 700°C in order to avoid the PZT crack problem if the ditch on the bottom electrode is wider than 3 μm. The top electrode deposited with second 200/20 nm Pt/Ti evaporation and lift-off procedure. The cantilever membrane is then created with a 445 μm KOH backside etch, and finally released with a 5 μm Si RIE. During the KOH backside etch, the front surface is covered with Apeizon wax to protect any KOH attack on the PZT layer.

3. Experimental Results and Discussion

The microstructure and crystal orientation of the PZT film were observed by a field emission type scanning electron microscope (FE-SEM) and X-ray diffraction (XRD). The microstructures and typical XRD pattern of the film are shown in Fig. 2. The PZT film is dense with approximately 100 nm grains and consists of randomly oriented perovskite phase structure.

The surfaces of the PZT film on the separated Pt bottom electrode region ("P" region in Fig. 1(a)) were observed at three points by an AFM (Atomic force microscope). Figures 3(a)–(c) show the surfaces of the PZT film directly on the 3 μm ditch, near on the 3 μm ditch, and on the Pt bottom electrode that are indicated as "a", "b" and "c" in Fig. 3(d), respectively. The surface morphologies are not the same at each region. The surface ot the PZT coated on the ditch between the Pt bottom electrodes is smoother and denser

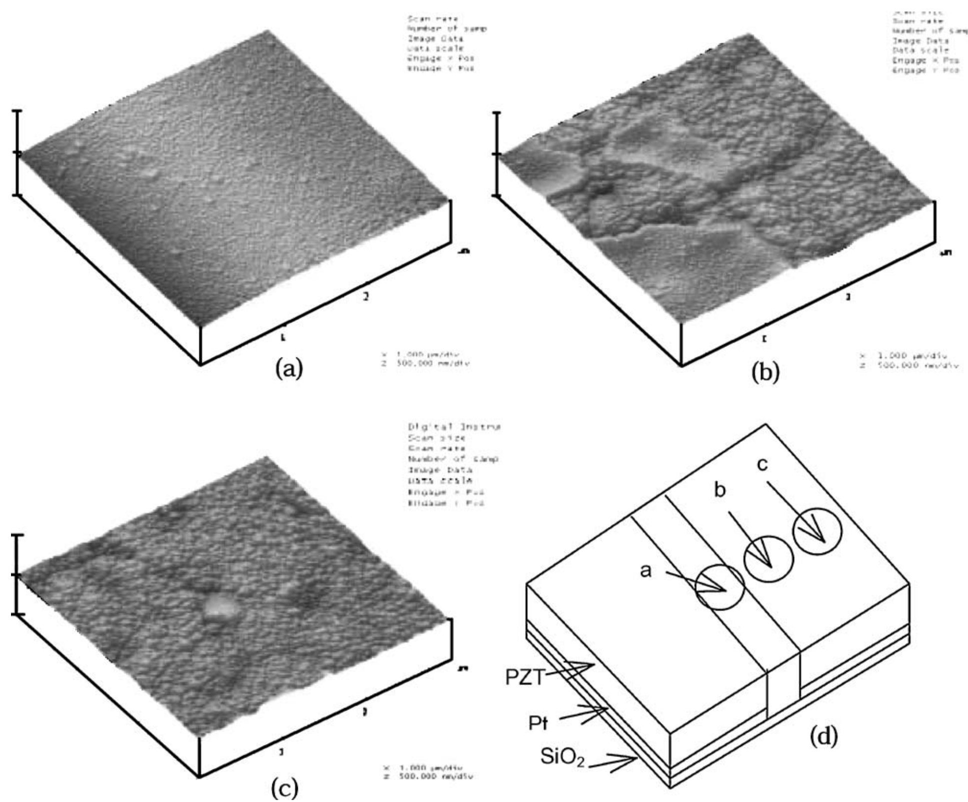
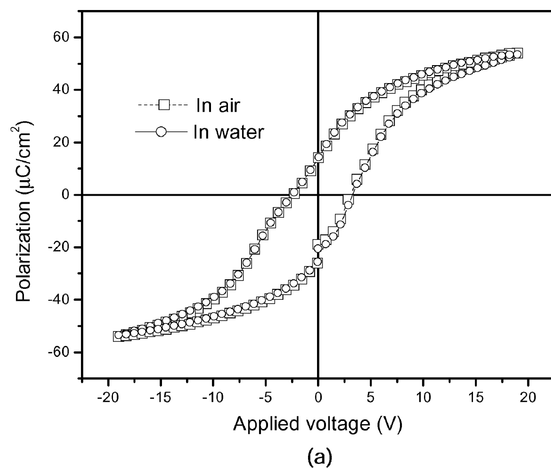


Fig. 3. AFM images of the PZT film surface and intersection diagram of the PZT film on the separated Pt bottom electrode region, (a) PZT surface directly on the separated Pt, (b) PZT surface near on the separated Pt, (c) PZT surface on Pt bottom electrode, (d) Schematic diagram of the region "P" in Fig. 1(a) which was observed by AFM.

(Fig. 3(a)). On the other hand the PZT surface on the Pt bottom electrode has submicron granular perovskite structure as shown Figs. 2 and 3(c). The different microstructures in each region is suspected to be due to the inter-diffusion between PZT and Si because the 200 nm silicon oxide is not sufficient to protect against PZT and Si inter-diffusion. The film on the ditch ("a" in Fig. 3(d)) should consist of insulating compounds such as $PbSiO_3$, $PbTiO_3$ and $ZrSiO_4$ according to the inter-diffusion between PZT and Si [5]. The surface of the inter-diffused new compounds is smooth and dense. We observed the PZT surfaces on the separated bottom electrodes with different distances of the separated gap. Ditch widths larger than 3 μm gaps tend to lend to cracks after annealing. The PZT film can also be etched before annealing at 700°C in order to avoid the PZT crack problem if the ditch width is than 3 μm. PZT layer is patterned by wet-etching process. The etch rate of the PZT film is approximately 0.25 μm/min with HCl: BOE (6:1): DI water (volume ratio of 100:16:200).

We have a test sample to measure and calculate the strain and piezoelectric constant. The structure is a double anchored actuator. The piezoelectric constant (d_{31}) of the PZT thin film, inferred from the measured displacement, is about −100 pC/N [6]. The dielectric polarization and fatigue properties of the immersible piezoelectric devices were measured in air and in water. To measure the property of the devices in water, a drop of water was supplied on the immersible region of the device continuously, and the polarization property was measured. It shows a possibility of water imersible actuator. Figure 4(a) shows the P-V hysteresis curves of the device. The spontaneous polarization (P_s), remanent polarization (P_r), coercive field and dielectric constant are almost the same values whether it is in air or in water. These property values are 54 μC/cm^2, 15 μC/cm^2, 60 kV/cm and 1200, respectively. The polarization property of the immersible PZT devices in the air was changed 28% from the initial value after 2×10^{10} cycles and failed after 3×10^{10} cycles of 19.6 μsec-rectangular pulses at 5 V. On the other hand, the device was changed 20% after 1.5×10^9 cycles and failed after 5.1×10^9 cycles in water (Fig. 4(b)).



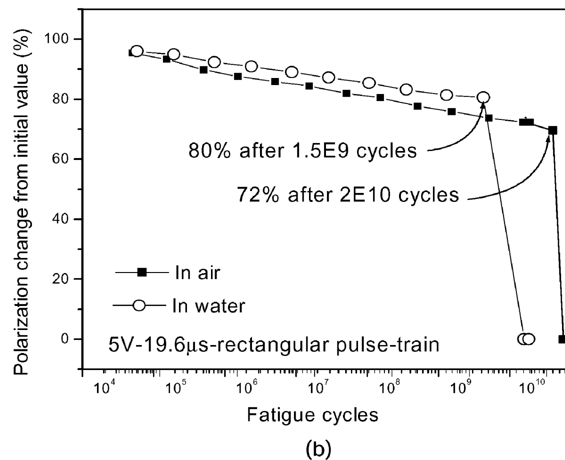


Fig. 4. Polarization property changes of the PZT films: (a) P-E hysteresis curve as deposition, (b) fatigue properties against number of cycles in air or water.

4. Conclusions

We have designed, fabricated, and demonstrated a water-immersible micromachined thin film piezoelectric actuator without any additional passivation layer. The fabricated piezoelectric PZT film shows excellent dielectric and piezoelectric properties such as spontaneous polarization (P_s), remanent polarization (P_r), coercive field, dielectric constant and piezoelectric constant (d_{31}). These properties are undifferentiated in air or in water, with values of 54 μC/cm^2, 15 μC/cm^2, 60 kV/cm, 1200 and −100 pC/N, respectively. The device permits fine fatigue characteristics in that the polarization property changes less than 20% after 1×10^9 continuous cycles in water. The device requires less than 3 μm of ditch width between the bottom electrodes to avoid PZT crack formation. These results present the feasibility of applications in underwater devices, such

as water-immersible pressure sensors or cantilever actuators for biomedical micro-devices. Future design iterations will optimize the separated bottom electrode geometry and improve device packaging to enhance the fatigue properties in water for long-term underwater operation.

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