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Microfabrication of PZT force sensors for minimally invasive surgical tools

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Abstract. Minimally invasive surgery (MIS) is the most exciting and rapidly developing area where force sensing is actually of central importance. Micromachined piezoelectric sensors can be integrated onto MIS tools for improved diagnosis and treatment monitoring. A micro-machined freestanding lead zirconate titanate (PZT) force sensor is fabricated using five masks process incorporating deep reactive ion, ion beam and wet-chemical etching techniques. The PZT sensor is designed as a parallel plate capacitor structure in which the sol-gel prepared 1-µm thick PZT film is sandwiched between top (Au/Cr) and bottom (Pt/Ti) metal electrodes mounted on a thin Si membrane. This paper also describes a new wet chemical approach for patterning $PbZr_{0.52}Ti_{0.48}O_3$ films. The etch recipe provided excellent etch control, minimized undercut, preserved the photoresist mask, and effectively removed the residues on the etched surfaces. A high etch rate (200 nm/min), high selectivity with respect to photoresist, and limited under-cutting (1.5:1, lateral : thickness) were obtained. The fabricated force sensor exhibited good ferroelectric properties. The current fabrication procedure and electrical analysis can be considered as a breakthrough for fabricating freestanding PZT force sensor in any desired shape and dimensions, as well as a good example of ferroelectric microdevices.

1. Introduction

Minimally invasive techniques enable surgical diagnostics and surgery to be performed through a small incision [1-2]. This greatly reduces the damage to healthy tissue leading to faster recovery times for the patient. However, the surgeon is deprived of the sensory feedback available with the currently available minimally invasive surgical (MIS) tools. Miniaturized sensors integrated onto the MIS tools can provide more information about location, diagnostics and treatment. Lead zirconate titanate (PZT) has been a well-known piezoelectric material widely used as sensors because of its large electromechanical coupling coefficients, high resistance to depolarization from mechanical stress and driving voltages [3]. These requirements are needed in applications such as biomedical (e.g., force, ultrasound, blood pressure sensors), manufacturing (e.g., microflow controllers, micro pumps, infrared detection and imaging), information processing (e.g.,displays, storage capacitors) and automotive (e.g., accelerometers) industries [4-10]. There have been many reports on piezoelectric sensors and actuators mostly with PZT bulk disks and thick passive structures of hundreds of micrometer in thickness. In addition, most of the structures were fabricated by bulk micromachining combined with additional assemblies such as manual gluing or wafer bonding. MEMS devices incorporating PZT films reported in literature are not very numerous due to difficulty in integration

and sensor design. There are few reports on diaphragm actuators using a few micrometer thick piezoelectric and passive layers which also generate deflections larger than the structural thickness [11-13].

However, all the above mentioned thick and thin film membrane PZT structures are supported on a Si substrate or micro-machined Si membranes and in the form of cantilever structures either single or two ends supported. That means that all these structures are always attached to the substrate and the substrate clamping effect in distorting the modes of displacements cannot be minimized. In the case of tactile sensor applications, PZT force sensor should be totally free from substrate, and the freestanding PZT force sensor leads the way for design flexibility and results in maximum force sensitivity in the range of mN to µN. The device characteristics of PZT based micro-sensors and micro-actuators used in biomedical MIS tools are very sensitive to micro-fabrication process. Etching the PZT thin film is an essential step in device fabrication process and has attracted considerable attention in recent years. Among the various dry etching processes, ion beam etching (IBE) is the most commonly used technique to pattern PZT thin films with no under cutting. However, because of the difference in sputtering efficiency of Pb, Zr and Ti, and the poor selectivity of PZT over photoresist (mask) and Pt (electrode), it is difficult to get satisfactory PZT patterns. The etch selectivity of PZT over photoresist and Pt is also found to be low in reactive ion etching (RIE) processes and even with high-density plasma assisted techniques such as inductively coupled plasma (ICP) and electron cyclotron resonance (ECR) it is difficult to get reliable PZT patterns. The aforementioned processes are also most suitable for films less than 1 micron thick because of the slow etch rate (100-320Å/min). However for thicker films these processes become extremely time consuming, expensive and cross-contamination is also a problem. Compared with dry etching, wet etching is an effective technique for MEMS due to its high etching rate, low cost and high selectivity [14-17].

Recently, many studies have been performed on patterning PZT and La doped-PZT (PLZT) films using wet-etching techniques [14-18]. In this paper, we propose a novel wet etch recipe to pattern PZT films grown on Pt coated Si substrates. In this work we used a combination of wet-etch, ion beam etching (IBE) and deep reactive ion etching (DRIE) processes to fabricate a free-standing PZT force sensors with top and bottom electrodes forming capacitor structure. This work demonstrates a micro-machined freestanding piezoelectric force sensor made of PZT thin film. This kind of force sensor can be applied to tactile sensing of minimally invasive surgical (MIS) tools, tactile skin for robotics to detect sub-mN forces.

2. Experimental

PZT thin films of composition PbZr_{0.52}Ti_{0.48}O₃ with 15 mol% excess Pb were prepared by a sol-gel method on Pt(150nm)/Ti(50nm)/SiO₂/Si substrates. A thin (50 nm) Pb_{1.05}La_{0.05}TiO₃ (PLT) layer was used as a seeding layer between PZT and Pt. After film deposition, photolithography with a two mask process (Mask-1 and Mask-2) was employed to define etching patterns of circular disks (diameters 1 to 2 mm) and cantilever beams (length (650 μ m), width (100 μ m)) using photoresist (AZP4620) and developer (AZ400K). A contact aligner (EVG 620) was used for ultra-violet light exposure. PZT films of desired thickness were spin-coated onto a cleaned Pt/Ti/SiO₂/Si wafer and annealed at 700°C for 1h. The details of film deposition is reported earlier [14-15]. The annealed PZT film was then spin-coated with a photoresist and soft backed at 110°C for 1 min. A set of circular and cantilever beam patterns were lithographically transferred onto the wafer using a white field Mask-1. The UV-exposed photoresist was stripped of in a developer mixed with DI-H₂O in the ratio 1:3. The developed photoresist pattern was used as mask to etch PZT in the chemical solution mixture or ion beam etching (IBE) process. The PZT etch solution consisted of {1 ml (BOE(6:1)) + 6ml (CH₃COOH) + 6ml (HNO₃(65%)) + 6ml (HCl (35%)) + 4g (NH₄Cl) + 2g (C₁₀H₁₈N₂O₁₀Na₂ (EDTA))} in 75ml H₂O

[14-18]. Once PZT was patterned, the photoresist could be easily removed by rinsing in N-methyl pyrolidone (NMP) at 85°C for 30 min.

After patterning the PZT with the first photolithographic mask, the top electrodes Au(100 nm)/Cr(50 nm) were e-beam evaporated and patterned using the dark field Mask-2 following a lift-off process using photoresist and NMP. IBE was used to pattern the bottom electrode after covering the PZT and top electrode with photoresist (Mask-3). Figure 1 illustrates the microfabrication sequence for the release of free-standing PZT force sensor. Two types of DRIE systems, namely DRIE-Si and DRIE-dielectric for etching silicon and SiO₂ were used. A SiO₂ layer of thickness ~ 0.5 μ m deposited using plasma enhanced chemical vapor deposition (PECVD) and patterned using DRIE-dielectric to create release trenches on the front side of the SOI wafer (Mask-4). Then DRIE-Si etching process was carried-out and stopped on the oxide. The release holes on the backside of the



Fig. 1. Fabrication sequence of the PZT force sensor release process.

SOI wafer was fabricated by making use of the Mask-5. The PECVD deposited SiO_2 and Si_3N_4 coatings of thickness ~ 2µm each were used as masking layers for DRIE-Si and wet-etch processes, respectively. First, DRIE-Si was used to remove the major part of the Si from the backside to about 480µm and the remaining Si ~ 20µm were removed by using wet-etching in tetramethyl ammonium hydroxide (TMAH) at 85°C. The wet-etch process enables a slow and controlled etching of the

remaining Si layer and the etch process could be comfortably stopped on the oxide, which eventually released the PZT force sensor from the SOI wafer.

Composition of the PZT films was measured by an energy-dispersive x-ray microanalyzer (EDX). The surface morphology was examined using an optical profiler (WYKO NT1100) and atomic force microscopy (AFM, Digital instruments, Nano Scope III). A Philips field emission (XL30 FEG) scanning electron microscope (SEM) was used to investigate the surface texture and the cross-section of the film. The phase formation and crystallinity of the films were analyzed using X-ray diffraction (XRD, X'pert Philips). The dielectric measurements were carried out at frequencies between 100Hz and 1MHz using a precision impedance analyzer (Agilent 4294A) with a signal voltage of 50 mV. The ferroelectric properties were measured with a precision workstation (Radiant Technologies). The I-V characteristics were evaluated using an HP4145B Semiconductor parameter analyzer.

3. Results and Discussion

3.1 Crystal structure and Surface Morphology

The XRD analysis of the multilayered PZT film of thickness ~ 1 μ m annealed at 700°C for 1h and patterned by a wet-etch or IBE process was carried out. The sharp diffraction peaks indicate better homogeneity and crystallization of films. All the films exhibited perovskite structures with random



Figure 2. (a) SEM micrograph of released freestanding PZT force sensor and (b) AFM surface morphologies of PZT force sensor after wet chemical etching.

orientations and dominant peaks corresponding to (110) and (100) reflections. There were no peaks corresponding to a residue formation on the etched patterns which indicate that the various components used to form the etching solution could successfully removed the residues. The EDX compositional analysis confirmed that the PZT films were of a stoichiometric composition of Pb/(Zr+Ti) ~ 1.0 and a Zr/Ti ratio of about 52/48. SEM analysis on the cross section of the PZT multiplayer film grown on the PLT layer and annealed at 700°C for 1 h shows that the grown film was dense and homogeneous. SEM analyses indicate that the interfaces between the film and substrate layers are well defined with a dense microstructure. Fig. 2(a) depicts the surface morphologies of the free-standing PZT force sensor. The overall thickness of the force sensor is ~ 11.3 μ m. The ratio between the amount of under-cut and the PZT thickness was found to be ~ 1.5:1 which is close to the ideal isotropic wet etch ratio of about 1:1. The surface morphologies of the films as examined by using an AFM (Fig. 2(b)) demonstrate that the microstructure consists of round and elongated grains

with considerable volume fractions of ultrafine grains, and the grain sizes are in the range of 40 to 120 nm. The root mean square (RMS) roughness of the multilayered film is ~ 20 nm.

3.2 Electrical Characterization

Fig. 3 demonstrates that the 1 μ m thick PZT structure displays a P_s of about 57 μ C/cm² at an applied electric field of 1000 kV/cm with P_r ~ 35 and E_c ~ 108 kV/cm at a charging time of 100ms. In comparison to PZT structures before release, the micro-fabricated free-standing PZT sensors exhibit a 40% reduction in P_s and P_r values which is profited by a 35% reduction in coercive field [14]. The reason for the reduction in ferroelectric parameters is not known, as EDX and XRD analysis didn't show any appreciable change in composition or phase of the released PZT structure. We believe that the various etching process may induce finer changes in the PZT microstructure and interface characteristics [14]. The other plausible reason could be minor change in the Ti concentration, due to its solubility in the wet etching solution consisting of BOE and HCl, which are potential Ti etchants.

A similar reduction in P_s (40 μ C/cm²), P_r (24 μ C/cm²) and E_c (42 kV/cm) values for wet-etch patterned PZT structures of thickness ~ 5.74 μ m were reported by Wang et al. [17] In comparison, the results presented here demonstrate higher P_r (35 μ C/cm²) and P_s (57 μ C/cm²) for 1 μ m thick PZT films.

The loss of switchable polarization or decrease in performance during operation with repeated polarization reversals that characterized ferroelectric fatigue in the PZT force sensor was carried out. At 10^{10} cycles, polarization loss of about 9-12% is observed in the PZT force sensor. The leakage current measurements demonstrated that the fabricated force



Fig. 3. Hysteresis loop of freestanding PZT force sensor

sensor showed a leakage of about 10^{-7} A/cm² at an applied electric field of 200 kV/cm and the current density increases to 6 x 10^{-5} A/cm² with further increasing the applied field to 1000 kV/cm. The relative dielectric constant and the loss tangent were measured as a function of frequency from 100 Hz to 100 kHz at room temperature for the PZT structure. The dielectric constant decreased steadily from ~ 800 to ~ 400 with increasing frequency from 100Hz to 100 kHz. The loss tangent varied from 0.04 to 0.06 in the above measured frequency range.

4. Conclusions

Successfully developed and fabricated a freestanding PZT force sensor using micromachining and wet etch processes. The PZT layers of thickness ~ 1 μ m were prepared by a sol-gel process. The microfabrication procedure was carried out using 5-masks process incorporating DRIE, IBE and wet etch techniques. The wet-etch process for patterning PZT provided excellent etch control, minimized the undercut, preserved the photoresist mask and effectively removed the residues on the etched surface. The PECVD deposited SiO₂ and Si₃N₄ layers served as excellent masking layers for the DRIE and TMAH etch processes and resulted in successful release of freestanding PZT force sensor (~11.3 μ m thick) with reliable electrical characteristics. The fabricated force sensor exhibited good

ferroelectric properties in terms of larger saturation polarization, P_s of ~ 57 μ C/cm² at an applied field of 1 MV/cm, higher remanent polarization, P_r of ~ 35 mC/cm², for a coercive field of ~ 75 kV/cm, fatigue free characteristics of up to $\geq 10^{10}$ switching cycles and a low leakage current density of 10^{-7} A/cm² at 200 kV/cm. Scope exists for fabricating the freestanding PZT force sensor in any desired shape and dimensions using microfabrication techniques. Presently developed PZT force sensor has the potential application possibilities in the areas of biomedical, robotics and micro-devices engineering.

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