Fabrication of High Quality PZT Thick Film Using Lift-Off Technique

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ABSTRACT

The present paper proposes a simple fabrication technique of the high quality lead-zirconate-titanate (PZT) thick films with a single coating and using a thick photoresist SU-8, which call for lift-off technique. The PZT films with the thickness of 100μm or higher were crack-free and had good morphology. The PZT films with perfect perovskite structure had excellent piezoelectric property and the d_{33} was about 170pC/N. Ferroelectric hysteresis loop was measured, and the remnant polarization (P_r) of the PZT film was about 25μC/cm^2 and the coercive field (E_C) was about 27kV/cm. In the radio-frequency (RF) region, the dielectric constant was about 350 and the dielectric loss was less than 0.01.

Keywords: PZT, Thick Film, Lift-off, SU-8, Piezoelectric

1 INTRODUCTION

PZT, a typical ferroelectrical material with outstanding dielectric, piezoelectric and ferroelectric properties, is expected to be widely used in nonvolatile ferroelectric random access memory (NVFRAM) and microelectro-mechanical system (MEMS). The use of PZT films rather than bulk ceramic technology can fully "integrated" device manufacture with the piezoelectric layer integrated directly onto a silicon substrate. In this way, there is the potential to increase manufacturing speed and decrease costs. For direct integration with silicon, the integration processes of silicon-based PZT films are the points.

The PZT films made from a sol-gel processing route have potential for using in a variety of electronic applications such as high-density capacitors, nonvolatile memories, sensors and actuators in micro-machined structures, and ultrasonic transducers. Although many PZT-based device applications require thin PZT films (less than 1μm), the demand for the thickness of PZT films used in MEMS is usually over 1μm due to the advantages of the thick thin films: larger surface displacement, higher or broader working frequency range and higher voltage sensitivity for micro-sensors.

In recent years, various methods of fabrication of PZT thick films have been proposed, such as tape casting [1], screen-printing [2], silicon mold process [3], sol-gel [4]. However, these methods have low compatibility with conventional IC/MEMS processes or cannot obtain enough large actuator forces, which is proportional to the thickness of the PZT film.

This paper proposes a simple fabrication technique of high quality PZT thick films with a single coating and using a thick photoresist SU-8, which call for lift-off technique. The preparation process of the PZT solution was improved. The simulataneous thermogravimetric (TGA) and differential thermogravimetric results (DTG) analysis of the PZT sol were measured. The PZT films obtained using single coating with the thickness of 100μm or higher, which was proportional to the thickness of the photoresist SU-8, were crack-free and had good morphology. Phase characterization and crystal orientation of the films were investigated by X-ray diffraction analysis (XRD). The piezoelectric constant d_{33} and the ferroelectric hysteresis loop of the PZT films were measured. The dielectric constant and the dielectric loss were obtained in the RF region.

2 PREPARATIONS OF PZT THICK FILMS

In the preparation process of the PZT solution, the choice of precursor compounds and solvents plays a major role. Lead acetate trihydrate, titanium iso-propoxide and zirconium n-propoxide are used as precursors. Propyl alcohol and acetic acid are used as the solvents. Figure 1 illustrates the flow diagram for the preparation of precursor solution.

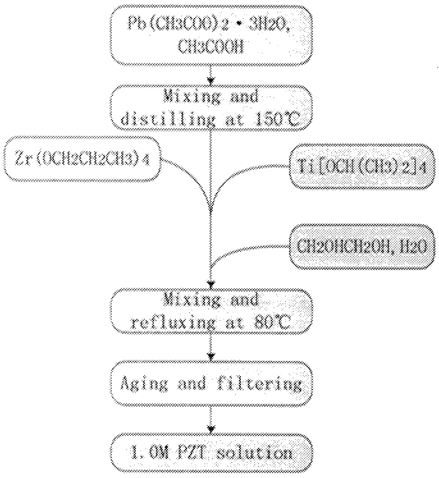

Figure 1: Preparation process of the PZT Solution.

Pt/Ti/SiO_2/Si multilayer substrates are adopted for PZT films deposition. The fabrication processes are shown in Figure 2.

1. Pt (150 nm) / Ti (27 nm) bottom layers are deposited on a silicon wafer (thickness: 380μm) using a vacuum deposition. An SU-8 100 layer is patterned to form the mold of the PZT films. The thickness of the SU-8 layer ranges from 75μm to 150μm.
2. The PZT solution is dispensed on the SU-8 layer. Then, the wafer with PZT sol is dried in a sealed container at room temperature to gelatinize the sol.
3. PZT wet gel is prebaked at 100-150℃ to remove the partial organic materials of the sol in the oxygen atmosphere.
4. The wafer is lapped using a urethane foam pad. Isopropyl alcohol (IPA) is used as solvent and lubricant. Excessive wet gel on the SU-8 layer is scraped off. This procedure prevents the gel in the SU-8 mold from cracking when the gel gets dry.
5. The gel is fired at 350℃ for 10 min in the oxygen atmosphere to remove the other organic materials. At this moment, the gel changes into amorphous solid of PZT, and the SU-8 layer separates from the PZT structures. Thus, SU-8 molds do not bond with the PZT structures while firing. Then, heat treatment is applied to the wafer.
6. Finally, the sample is annealed at 650-700℃ for 5min using the rapid thermal annealing (RTA) to crystallize them into a perovskite structure.

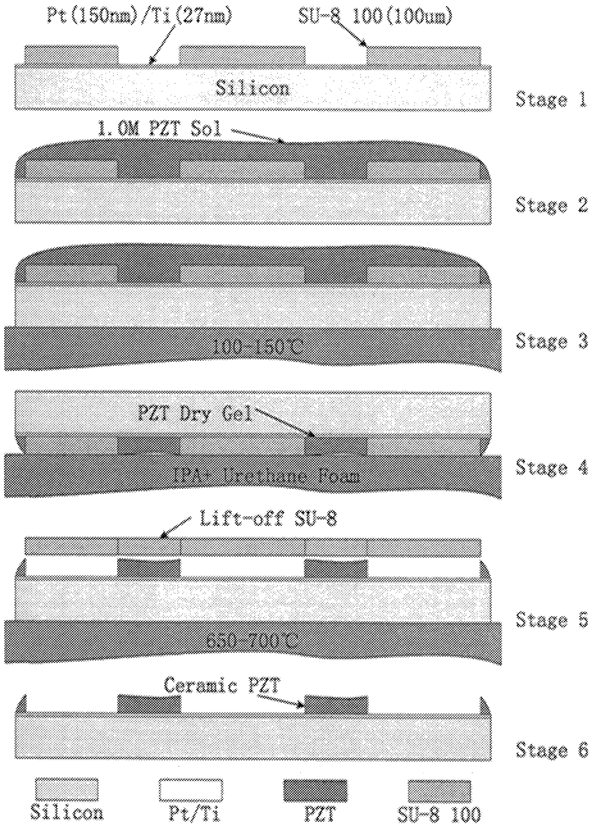

Figure 2: Fabrication processes of the PZT thick films.

3 CHARACTERISTICS OF PZT THICK FILMS

The TGA and differential thermal analyses (DTA) are completed using a Netzsch STA429 thermal analyzer on the PZT sol. The TGA results in Figure 3 show weight loss versus temperature. Most of the weight loss occurs below the temperature of approximately 350℃, with no further weight loss past 400℃. The DTG results in Figure 3 combine the results of the TGA and DTA analyses to show rate of weight loss versus temperature for the same PZT sol dried at a temperature of 150℃ prior to TGA/DTA analysis. An important point is the decomposition temperature of PZT gel at 200℃ at which the rate of weight loss peaks. The rate of weight loss increases again at higher temperatures due to the pyrolysis of the remaining organic components. On the basis of this data, a thermal processing schedule was developed to ensure that the organic components of the film are released in a controlled manner throughout the process. These temperatures and times are important in the development of the fabrication process toward releasing all the organic components from the film in a controlled manner to reduce film stress, avoiding the formation of the non-piezoelectric PZT pyrochlore phase and fully crystallizing the film into the piezoelectric perovskite phase.

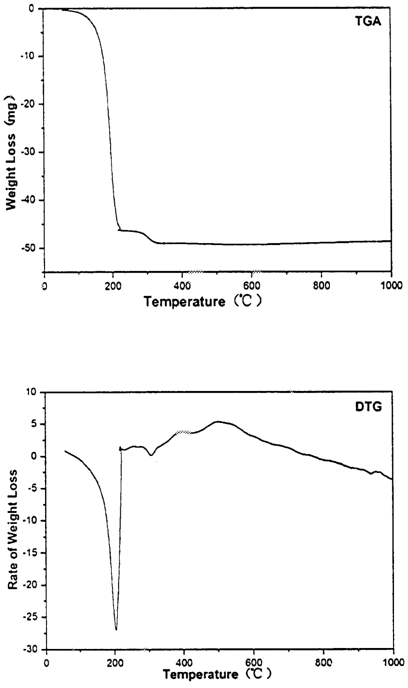

Figure 3: TGA and DTG analysis of PZT sol.

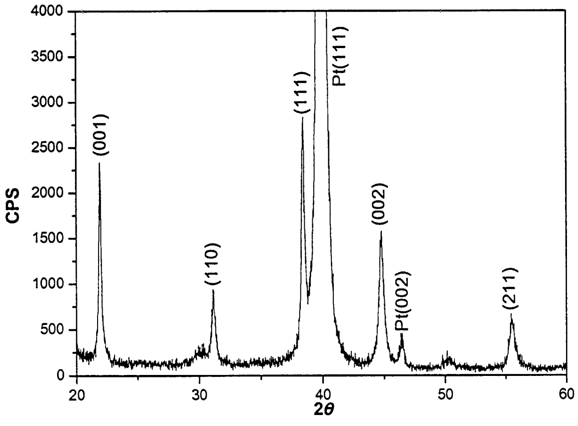

Figure 4: XRD pattern of the fabrication PZT.

Figure 4 shows the XRD pattern of the phase characterization of the PZT film. From the pattern, the sample exhibits two strong peaks at $2\theta = 22^\circ$, $2\theta = 38^\circ$ corresponding to the (001) and (111) peaks. Other peaks of PZT perovskite structure can also be observed at $2\theta = 31^\circ$, $2\theta = 45^\circ$ and $2\theta = 55^\circ$, respectively. It is indicated that the sol-gel derived PZT films are crystallized completely.

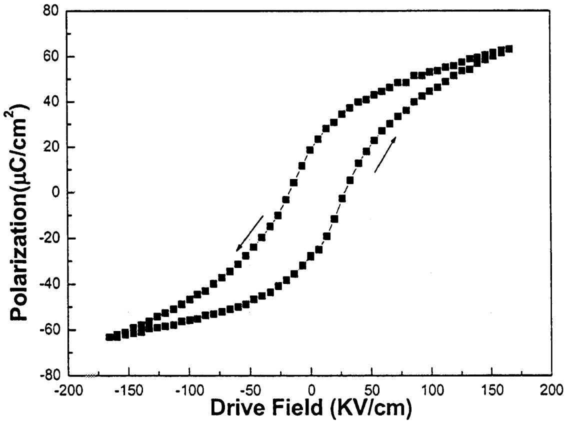

Figure 5: Ferroelectric hysteresis loop of PZT films.

As is known, the spontaneous polarization of ferroelectric materials does not follow the electric field linearly, and thus the polarization-electric field (P-E) hysteresis loop was resulted. Figure 5 shows the ferroelectric hysteresis loop of the PZT films. The P_r of the PZT ceramic is about 25μC/cm^2 and the coercive field E_C is about 27kV/cm.

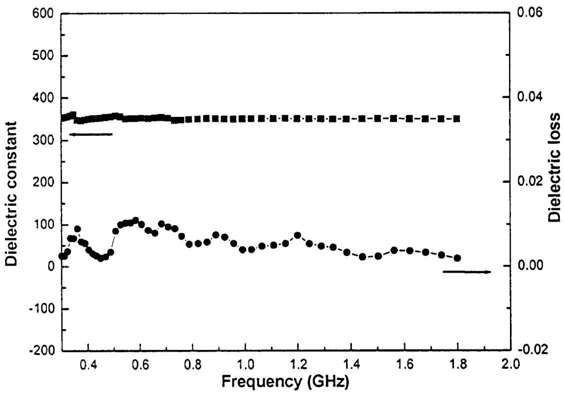

Figure 6: Dielectric responses of PZT thick films.

The dielectric responses of the PZT films, examined in terms of the dielectric constant and dielectric loss as functions of measuring frequency, are shown in Figure 6. For the PZT films, the dielectric constant changes very slowly as the frequency is above 300MHz. Known from Figure 6, the dielectric loss decreases continuously as the frequency increases, and is lower than 0.01 as the frequency is above 300MHz.

The experimental results show that the PZT films have excellent high-frequency properties: high dielectric constant and low dielectric loss. So, PZT is a promising candidate for applications in RF MEMS and microwave communication systems to meet the requirements for both high isolation and small device size, because the device size and the quality factor (Q value) are inverse proportional to $\sqrt{\varepsilon_r}$ and the dielectric loss, respectively.

The measure of the piezoelectric constant d_{33} shows that the PZT films have good piezoelectric property and the d_{33} is about 170pC/N and can be suitable for the applications of force-electric coupling devices.

4 CONCLUSIONS

The present paper proposes a simple fabrication technique of high quality PZT thick films with a single coat and using a thick photoresist SU-8, which call for lift-off technique. The TGA and DTG analysis of the PZT sol are measured. The PZT films obtained using single coating with the thickness of 100μm or higher, which is proportional to the thickness of the photoresist SU-8, are crack-free and have good morphology. The PZT films with perfect perovskite structure have excellent piezoelectric property and the d_{33} is about 170pC/N. Ferroelectric hysteresis loops are measured, and the P_r of the PZT is about 25μC/cm^2 and the E_C is about 27kV/cm. In the RF region, the dielectric constant is about 350 and the dielectric loss is less than 0.01.

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