Optimised Piezoelectric PZT Thin Film Production on 8'' Silicon Wafers for Micromechanical Applications

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ABSTRACT

Piezoelectric materials are of great interest for new MEMS devices for sensors, actuators and energy harvesting. Due to its large electromechanical coupling coefficient Lead Zirconate Titanate (PZT) is one of the most favourable materials for realisation of fast and energy efficient microactuators. Here we report results of process optimisation for high quality thin film PZT deposition using standard magnetron sputter equipment (Oerlikon Balzers) of up to 2µm thickness at a deposition rate of 45nm/min. A reliable industrial production process was developed leading to optimised material quality for micromechanical applications. High quality films have been prepared on standard 8" silicon wafers using an Oerlikon CLN 200 tool with heated substrate holder. Ti/Pt bottom electrode configuration, deposition temperatures and process control have been varied for optimisation of thin film piezoelectric coefficients. Using Electron Probe Micro Analysis (EPMA) and X-ray diffraction (XRD) it was determined that best material composition and crystalline structure was achieved using a wafer chuck temperature of 600º C. Optimised PZT films of various thicknesses show a high dielectric constant ε_r of about 1500. A remarkable high average transversal piezoelectric module $e_{31,f}$ of -17,3C/m² was achieved. An effective longitudinal $d_{33,f}$ coefficient of 160pm/V was determined for 2µm thick PZT films. Using the optimised magnetron sputter process maximum values for the transversal piezoelectric module $e_{31,f}$ of -21C/m² have been determined.

Top electrodes on PZT thin films were realised in Cr/Au. These electrodes were used as hard masks for structuring of the piezoelectric material. Free standing PZT beams were produced by etch removal of a sacrificial layer below the PZT films. Various designs have been realised. Process flow and design examples for new sound transducers in the sound and ultrasound region are presented and discussed.

Keywords: MEMS, actuator, PZT, magnetron sputtering, sound transducer

1 INTRODUCTION

Piezoelectric materials are promising candidates for powerful actuators in MEMS devices [1], [2], [3]. The large electromechanical coupling coefficient of Lead Zirconate Titanate ($Pb(Zr_xTi_{1-x})O_3$, PZT) allows the realisation of actuators at high frequencies with low operation voltage and low energy consumption. In the same way large electromechanical coupling coefficients lead to high efficiency in energy conversion in sensor and energy harvesting applications.

Despite the fact that the deposition of PZT thin films is a field of intensive research, the fabrication of thin films is still quite challenging. Common thin film deposition techniques like MOCVD [2], magnetron sputtering [3] and Sol-Gel [4] suffer from low deposition rates and therefore are typically applied for the deposition of layers in or below micron range. The recently described method of sol hydrodynamic deposition [5] can produce surface structures of more than 30µm thickness but these structures are limited to small spots and are not suited for formation of a thin film. To improve PZT deposition rate for thin film application Fraunhofer ISIT has developed a gas flow sputter technique with a deposition rate of 100-120nm/min for high quality PZT films [6], [7], [8]. With this technique film thicknesses of up to 10 micron have been deposited on 8" wafers. Magnetron sputtering on the other hand allows utilisation of standard sputter equipment compatible with process equipment in semiconductor and MEMS production lines. Here we report results of an optimised magnetron sputter process using an Oerlikon CLN 200 sputter tool. A deposition rate of 45nm/min was achieved for deposition of high quality PZT thin films. To evaluate material quality films with up to 2 micron thickness have been fabricated and characterized.

In the following results of process optimisation and PZT thin film parameters are presented. For production of MEMS devices PZT films and the substrate below the PZT films have to be processed. Realisation of an initial process flow and first PZT actuated MEMS devices are discussed.

2 EXPERIMENTAL

For subsequent sputtering of PZT thin films, a Pt bottom electrode was used. The metallic film was deposited on (100) oriented 8” silicon substrates passivated with a thermally grown silicon oxide of 200nm and 1000nm thicknesses. Electrodes were directly prepared at ISIT by evaporating a bi-layer stack of 20nm Ti / 100nm Pt. To improve the crystallization of Pt and the forming of a distinctive (111) texture, a post-anneal was performed in a furnace at a pressure of about 1µbar and 550°C. Various electrode and seed layer configurations were evaluated for process optimization [9].

1000nm thick PZT thin films were grown by an in-situ RF magnetron sputtering process on an 8-inch capable Oerlikon Clusterline 200 tool. The film material is sputtered from a 300 mm ceramic target with an excess of lead oxide and a Zr/(Zr+Ti) ratio of 53/47 that corresponds to the morphotropic phase boundary composition of PZT. The films were deposited at chuck temperatures of 550°C, 600°C and 70 °C, a sputter power of 2 kW and a constant process pressure of $2{,}7\cdot10^{-3}$ mbar. For calibrating the actual temperature of the substrate, a silicon sense wafer wired with thermocouples was placed and heated on top of the used wafer holder inside the sputter tool.

For the piezoelectric and electrical characterization a Cr/Au top layer was deposited and patterned to fabricate capacitor structures with a specified top electrode layout.

The chemical and crystallographic properties were investigated by **E**lectron **P**robe **M**icro**A**nalysis (EPMA) and X-ray diffraction (XRD) with Cu Kα radiation. The quality of crystal orientation was evaluated from the FWHM of X-ray rocking curves.

For the determination of the $e_{31,f}$ coefficients samples were deformed in a 4 point bending system generating a defined uniform strain in the thin PZT film [10]. The $e_{31,f}$ coefficient can be easily calculated from the generated charges and geometrical and material properties of the substrate. The $d_{33,f}$ coefficient is determined by a precisely measuring double beam laser interferometer (DBLI) tool that eliminates the influence of the substrate bending [10]. Both tools are certified measurement systems from aixACCT systems GmbH [10].

3 RESULTS

To achieve the intended chemical composition of the PZT thin films, the influence of the sputter deposition temperature has been investigated experimentally in its dependence from the applied chuck temperature using various set points between 550 °C and 700 °C using various substrate modifications [9].

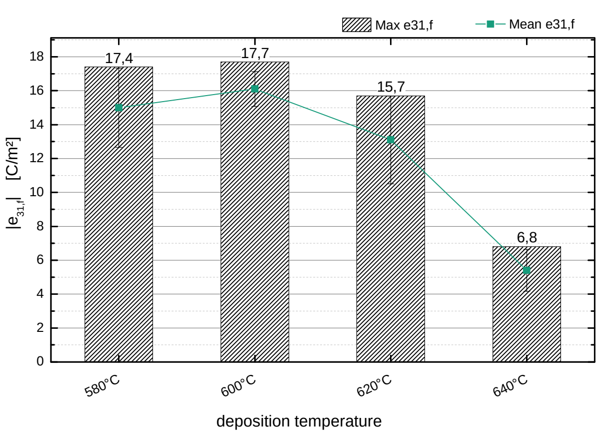

Fig. 1: Mean value of $e_{31,f}$ coefficient prepared at various chuck temperatures.

Resulting PZT material composition and thin film material quality has been discussed and presented in Ref. [9]. Optimum piezoelectric coefficients and material quality were obtained at a chuck temperature of 600ºC (Fig. 1).

In addition to the chemical composition the crystal structure of the deposited films has been investigated by XRD. At chuck temperatures of 550 °C beside a perovskite phase a considerable amount of a meta-stable non-piezoelectric phase becomes clearly visible. The preferred formation of this phase instead of the perovskite phase is attributed to an insufficient substrate temperature at these process conditions.

On the other side chuck temperatures of 700 °C lead again to strong signals of the non-piezoelectric phase when an evaporated Ti/Pt bottom electrode was coated with PZT. Due to the high deposition temperature the observed lead deficiency probably causes a stabilisation of the pyrochlore phase as previously reported by Kumar et al. [12].

Material parameters of optimised PZT films are summarized in Fig. 2 and Tab. 1.

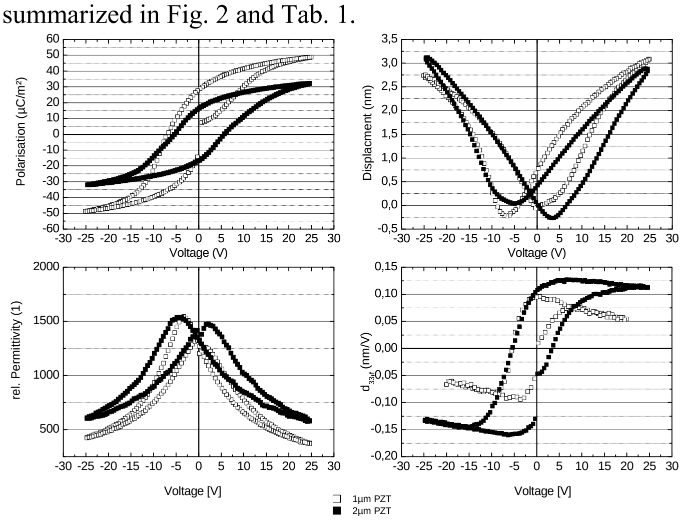

Figure 2: Material parameters of optimised 1 micron and 2 micron thick PZT films on silicon.

	large signal excitation					
film thickness	$\lvert d_{33,f}\rvert$ @ 25V	P_{r+}	P_{r-}	P_{max}	V_{c+}	V_{c-}
nm	pm/V	µC/cm²	µC/cm²	µC/cm²	V	V
1000	123	28,1	-12,7	48,7	0,3	-6,9
2000	129	16,2	-17,0	32,0	5,7	-5,3
	small signal excitation				mechanical excitation	
film thickness	$\lvert d_{33,f}\rvert$	max. rel. permittivity			$e_{31,f}$	
nm	pm/V	-			C/m²	
1000	95,9	1544			-17,0	
2000	159,4	1536			-17,3	

Tab. 1: Material Parameters of 1µm and 2µm thick PZT films on silicon, deposited at a chuck temperature of 600ºC.

Experimental trials were performed to optimize piezoelectric parameters. Process optimization to improve deposition uniformity on 8" wafers is not yet done. Fig. 3. (top) shows distribution of $e_{31,f}$ values on an 8" wafer and Fig. 3. (bottom) $d_{33,f}$ values on the same wafer.

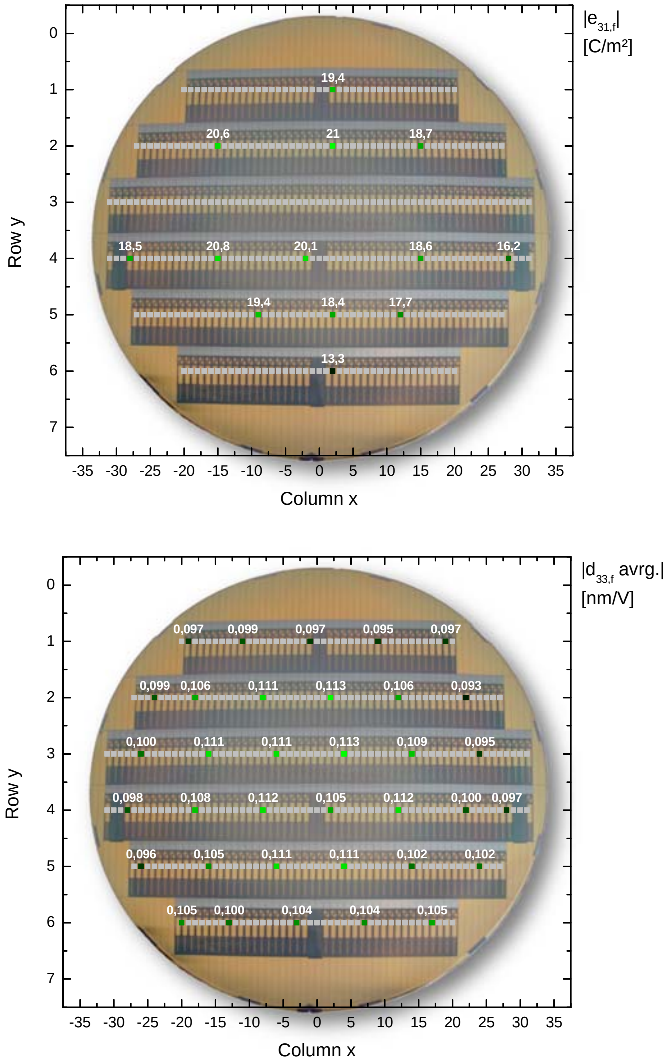

Figure 3: Distribution of e31,f (top) and d33,f (bottom) piezoelectric coefficients on 8" silicon wafer.

3.1 Process development for piezoelectric microactuators

To integrate sensing or actuating structures into a MEMS device in many cases the PZT films must be structured. This will be done by pattern definition by photolithography followed by an etching process. Experiments have been performed to evaluate wet and dry etch processes for PZT. For patterning with a wet etch process a wet chemical etching mixture consisting of HCl and HF can be used [11]. For a MEMS device additional process steps are necessary like deposition of functional layers, patterning of functional layer and top electrodes and finally release etch of mechanical structures. Standard photo resists show poor quality in etch processes for pattern transfer into a PZT layer.

Thus the photo resist was replaced by a Cr/Au metal layer which was previously patterned in a wet etching process. The good adhesion of the metal hard mask on top of the PZT allows an almost perfect isotropic etching of the PZT and leads to a drastically reduction in the under etching of the PZT layer.

For a more anisotropic pattern transfer a DRIE dry etch process using an advanced plasma system (APS) tool of the company STS was used on 8" silicon wafers with 2µm PZT using an SF_6 chemistry. Since Pb compounds are in general in volatile the selective chemical part of the DRIE etch process is limited. Hence the main process is more a physical sputter process while the chemistry is adjusted to increase selectivity over any given mask.

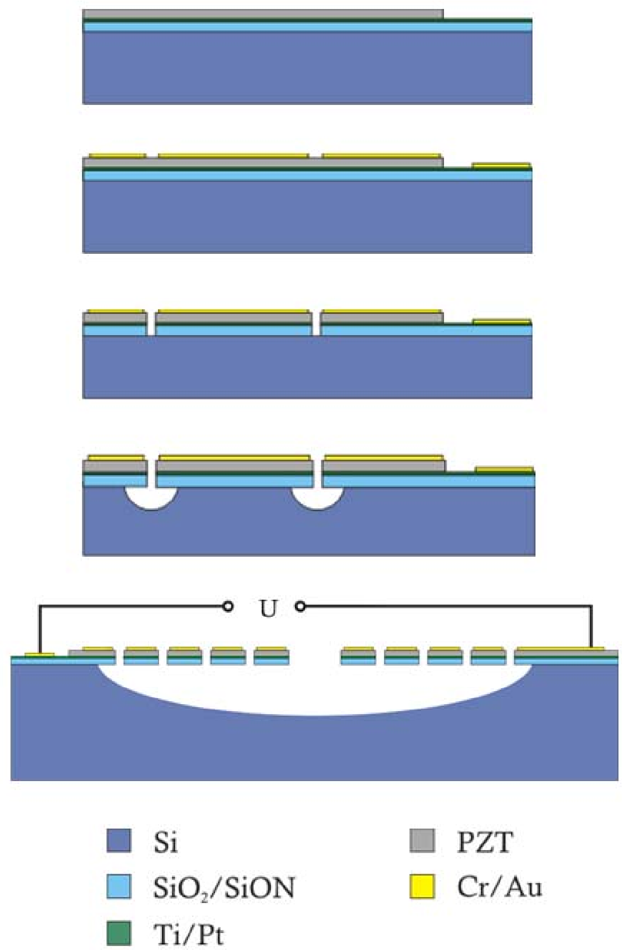

Figure 4: Process flow for PZT actuated MEMS devices.

First MEMS devices have been realized using the following process flow: - deposition of SiO_2 and SiON on a silicon wafer as functional layer, - evaporation of Ti/Pt bottom electrode, - PZT deposition by optimized magnetron sputtering, - dry and wet etch of PZT, - deposition of Cr/Au top electrode, - wet etch Cr/Au, - dry etch of PZT, Ti/Pt, SiON/SiO_2 ,- Si release etch with XeF_2. First design examples have been manufactured using the described process (Fig. 5).

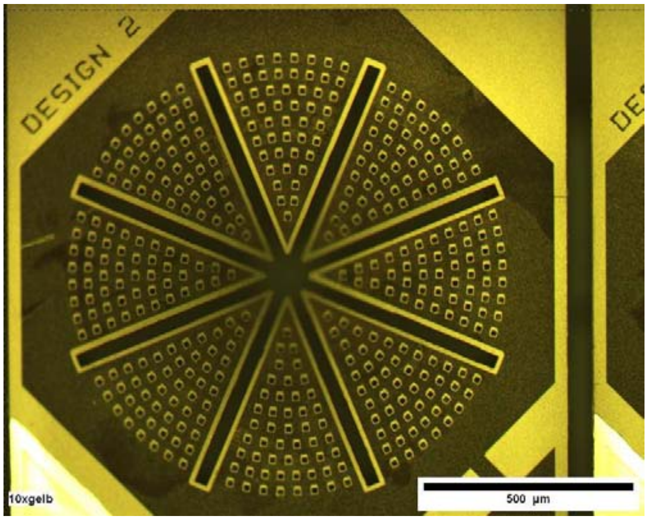

Fig. 5: Design example for a sound transducer using a PZT MEMS production process.

The device shown in Fig. 5 consists of eight triangular shaped free standing beams that can be bend by voltage application. The electromechanical properties of the novel device are under investigation. After release etch the moveable beams bend upwards by 45 µm between frame and center of the structure due to the residual stress in the functional SiON/SiO_2, Ti/Pt, PZT, Cr/Au layer system.

4. SUMMARY

1000nm and 2000nm thick PZT films have been deposited on 8-inch wafers prepared with sputtered and evaporated Pt electrodes at chuck temperatures of 550°C, 600°C and 700°C by RF magnetron sputtering. Films in-situ deposited at 550 °C exhibit a high content of non-piezoelectric pyrochlore type phases, due to an insufficient deposition temperature. Purely perovskite phase of the PZT is achieved at an applied chuck temperature of 600°C on sputtered Ti/Pt electrodes whereas at a chuck temperature of 700°C the amount of secondary phases rises again. A deposition rate of 45nm/min is achieved by magnetron sputtering.

From test samples an $e_{31,f}$ value of up to -21C/m² can be determined using an optimised magnetron sputter process.

For the patterning of PZT films wet and dry etch processes have been evaluated. A dry etch process could be identified with sufficient resist : PZT selectivity and good uniformity over an 8" wafer.

The electromechanical properties of PZT films prepared by magnetron sputtering at elevated temperatures are further investigated and compared to material parameters of material deposited by gas flow sputtering. Initial processes for device production of PZT actuated MEMS devices have been realised. Work is in progress for optimization and stabilization of the PZT deposition process. to increase the piezoelectric coefficient $d_{33,f}$ and $d_{31,f}$ respectively and to identify the best application areas for various PZT materials in MEMS devices.

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