Deposition of functional PZT Films as Actuators in MEMS Devices by High Rate Sputtering

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ABSTRACT

Crack and void free polycrystalline Lead Zirconate Titanate (PZT) thin films in the range of 5 µm to 15 µm have been successfully deposited on silicon substrates using a novel high rate sputtering process. The sputtered PZT layers show a high dielectric constant ε_r between 1000 and 1800 and a distinct ferroelectric hysteresis loop with a remanent polarisation of 17 µC/cm^2 and a coercive field strength of 5.4 kV/mm. A value of $d_{33,f}$ = 80 pm/V for the piezoelectric coefficient has been measured.

Based on this deposition process a membrane actuator consisting of a SOI layer and a sputtered PZT thin film was prepared. The deflection of this membrane actuator depending on the driving voltage was measured with a white light interferometer and compared to the results of **f**inite **e**lement **a**nalysis (FEA). With this approach a transverse piezoelectric coefficient e_{31} = -11 C/m^2 was approximated.

Keywords: MEMS, actuator, PZT, gas flow sputtering

1 INTRODUCTION

Piezoelectric materials are promising candidates for powerful actuators in MEMS devices. 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 and low energy consumption. In order to design large force MEMS actuators high quality PZT films with a thickness in the order of 10 µm are demanded.

Despite the fact that the deposition of PZT thin films is a field of intensive research, the fabrication of thicker films is still quite challenging. Common thin film deposition techniques like MOCVD [1], magnetron sputtering [2] and Sol-Gel [3] suffer from low deposition rates and therefore are typically applied for the deposition of layers in or below micron range. Screen printing methods [4] suffer from high firing temperatures above 900°C.

Results are presented for a new high rate sputter process for the deposition of high quality PZT films with a thickness of 5µm to 15µm. As a first demonstrator for a MEMS actuator a PZT film has been produced on top of a silicon membrane produced by a standard KOH wet etch process on a SOI wafer.

2 EXPERIMENTAL

The gas flow sputtering technique is a special physical vapour deposition (PVD) technique, based on a hollow cathode glow discharge and a gas flow driven material transport. In contrast to magnetron sputtering, the target is hollow and arranged perpendicular to the substrate. In this work a circular sputter source with a diameter of 40mm has been used. Therefore the substrate holder was moved linearly to increase the coated area and to improve uniformity of deposited PZT films on the wafer. Experiments have started to exchange the cylindrical sputter source with a set up of parallel sputter plates of 250mm width. With this improved sputter system standard 8" Si-wafers will be processed.

An argon flow through the sputter source transports the eroded metal atoms to the substrate where oxygen is added separately. For deposition of the PZT films pure metallic targets are simultaneously sputtered leading to the desired high sputter rate. The working pressure of 0,5 mbar inside the vacuum chamber for the hollow cathode discharge is maintained by a roots blower unit and a vacuum rotary pump. The gas flow sputter technique is described in more detail elsewhere [5, 6]. Deposition rates of 200-250nm/min have been achieved.

Deposition of ternary or quaternary films by reactive PVD is complicated due to strong differences in the vapour pressure of the elements, their reactivity or by their inmiscibility making target manufacturing impossible. In such cases, gas flow sputtering has strong advantages since targets composed of segments from pure elements can be used. The segments should be arranged along the gas flow direction resulting in a thorough mixing of the sputtered material during gas transport. Changing the size of individual segments, the film stoichiometry can be adjusted. This saves much time compared to experiments using alloy targets. In this work the target was composed of individual metal rings of lead, zirconium, and titanium of appropriate thickness. Oxygen is fed into the space between source and the substrate becoming activated there by the hollow cathode plasma.

During sputtering, the substrates are mounted on a heated wafer holder (chuck), where a bias-voltage is applied. The wafer holder allows substrate temperatures of up to 700 °C during sputtering. In order to obtain the

desired crystallographic microstructure of the PZT films, the deposition temperature is of major importance. Experiments showed that a minimum temperature of 550°C is necessary to initiate a crystalline growth of the PZT film, while temperatures of approx. 620°C are required for the deposition of PZT films with good piezoelectric properties. Typical process parameters are summarised in table 1 [7].

Substrate temperature	550–650 ◦C
Pressure	0.4–0.7 mbar
Argon flow rate	800 sccm
Oxygen flow rate	20 sccm
Source power	600W
Source voltage	400–600V
Bias AC voltage	50–100V at 200 kHz

Table1: typical values of main process parameters

2.1 Material Composition

A crucial aspect in sputtering compound materials consisting of various elements is the stability and reproducibility of the chemical composition of the deposited thin films. Since the piezoelectric properties of PZT films depend very strictly on their exact stoichiometry the repeatability of the composition in the process becomes even more important.

In fact many process parameters influence the stoichiometry like the used power level, target geometry, gas flows and substrate temperature. Therefore process control and optimisation are of high importance.

Material compositions of finished PZT films were determined by electron probe micro analysis (EPMA). While EPMA allows material analysis of PZT films after preparation other techniques have to be used to monitor the material composition during the deposition process. Optical emission spectroscopy (OES) gives an indication of relative material content within the glow discharge during sputtering. Therefore OES was used to monitor the sputtering process and control process uniformity by regulating Ar flow and RF power. The emission spectra were recorded by a grating spectrometer with a sensitive CCD detector (ARC, FC 459180). In several experiments it was verified that the relative emission intensity between the Pb emission line at 406nm and Ti emission lines around 399nm is linearly related to the Pb/Ti ratio in the deposited films. This linearity holds in the vicinity of the operational regime that was adjusted for the sputtering process here. Emission lines from Zr were too weak and noisy to be used as reliable control signal. Therefore OES was used for an optimised process control in PZT deposition leading to improved stability and reproducibility of the sputter process of PZT layers.

3 RESULTS

Thickness of PZT films was determined by SEM (fig. 1). Deposition rates of 200-250 nm/min were observed. These high sputter rates are about 20 - 25 times higher than reported from reactive magnetron sputtering [2] and demonstrate the potential of this sputtering technique. Typical layers had a thickness of 6 µm sputtered in about 30 min. However, even 16 µm thick PZT films were deposited without cracking or delamination within 90 min.
Electrical measurements of the relative permittivity ε_r have shown values between 1000 and 1800. A distinct ferroelectric hysteresis loop with a remanent polarisation of 17 $\mu C/cm^2$ and a coercive field strength of 5.4 kV/mm has been observed which is comparable to those of screen printed PZT [5]. Measurements of the piezoelectric coefficient $d_{33,f}$ using a double-beam interferometer [8] have shown values of the reverse effect of up to 79.7 pm/V.

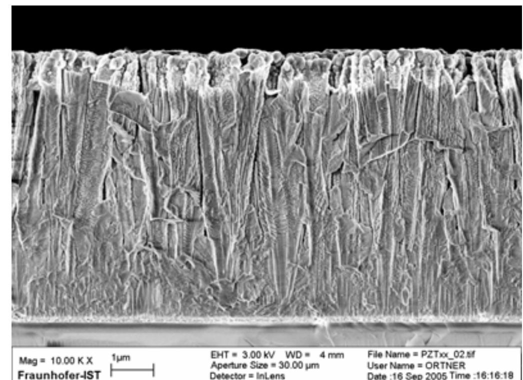

Figure 1: SEM cross section of a 6.6µm thick PZT film

3.1 Membrane actuator

To demonstrate the functionality of the PZT films, a membrane actuator was fabricated with a silicon MEMS process flow. The membrane actuator consists of the active, piezoelectric PZT film with some inter-layers on top of an elastic silicon layer fabricated from a silicon on insulator (SOI) wafer (see fig. 2). The membrane has a quadratic shape with a side length of about 2 mm.

This lateral dimension results in relatively large centre deflections of the membranes thus allowing the use of a commercial white–light interferometer (ATOS Micromap) to measure the bending of the actuated membrane.

The quadratic shape of the membrane is caused by the use of an anisotropic wet etchant (KOH solution, 30%, 80°C) during the membrane etching process. The principle design of the membrane is summarised in figure 2.

After PZT film deposition the PZT material is structured by lithography and wet etching to restrict the actuator material to the position above the membrane structure.

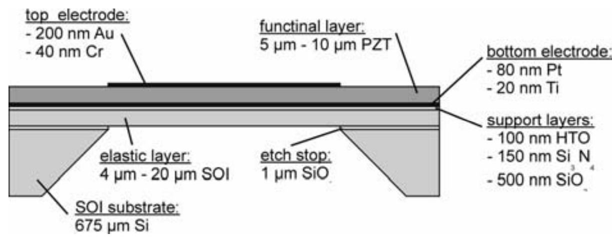

Figure 2: Schematic view of the unimorph membrane actuator.

3.1 Process Flow

150 mm SOI-Wafers with a SOI - thickness of about 20µm and an overall thickness of 675µm are used as substrates. In a first process step a diffusion barrier of 500 nm thermal oxide, 150nm Si_3N_4 and 100nm **H**igh **T**emperature **O**xide (HTO, SiO_2) is deposited in a **l**ow-**p**ressure **c**hemical **v**apour **d**eposition (LPCVD) process. Because of its small lattice mismatch to PZT and its inert behaviour Platinum is used as bottom electrode. To improve the adhesion of Platinum it is recommended [10] to use Titanium as adhesion layer. Both thin films were deposited by e-beam evaporation (Unaxis, BAK). To improve the texture of the Platinum layer the wafers are annealed at 550 °C in a vacuum oven for 30 minutes at a pressure of less than 0.1 Pa. This process leads to a complete (111) texture of the Platinum film.

Afterwards, these wafers are sputter coated with a PZT thin film of approx. 4 -5 µm thickness [5]. For patterning the PZT thin film a wet chemical etching mixture consisting of HCl and HF is used [9].

Tests ended up with an etch rate of the PZT between 1.3µm/min and 1.6µm/min leading to a total process time of approx. 4 - 5 min including over-etching. Due to the HF content and the required etching time the photoresist shows strong delamination effects leading to a poor quality in transferring the pattern into the PZT layer.

Thus in further experiments the photoresist was replaced by **p**lasma **e**nhanced **c**hemical **v**apour **d**eposition (PECVD) silicon nitride hard mask which was previously patterned in a dry etching process. The good adhesion of the Si_3N_4 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 to approx. 5 µm, see figure 3.

Finally the remaining silicon nitride layer is removed by dry etching. Subsequently a top layer is formed by local electroplating of gold with a thickness of 1.8 µm on a plating base consisting of 40 nm Chromium and 200 nm Gold. After removing the plating base the silicon nitride passivation on the rear side of the wafer is patterned by dry etching. This silicon nitride layer acts as hard mask during the deep anisotropic wet etching in KOH solution (fig. 4). Finally the silicon oxide beneath the SOI layer is removed in a HF vapour etch process.

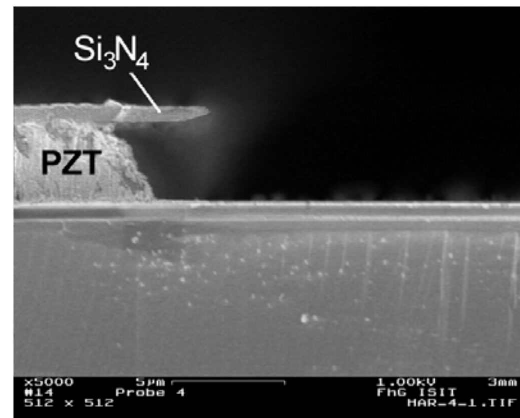

Figure 3: SEM cross section of a 5µm thick PZT layer etched by isotropic wet etching using a Si_3N_4 hard mask.

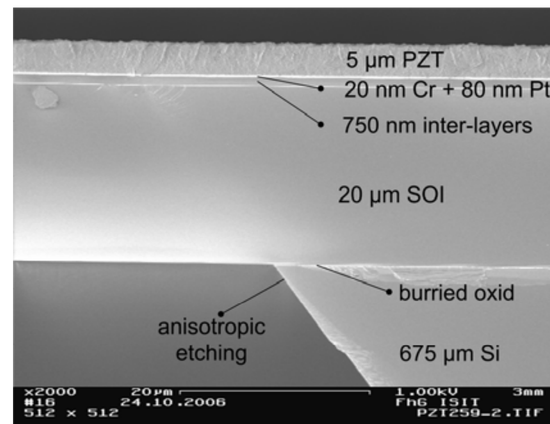

Figure 4: SEM cross section of a membrane: on top of 20 µm silicon (SOI) a stack of 750 nm diffusion barrier layer can been seen. Beneath the PZT layer with a thickness of approx. 5µm the metal layers consisting of 20 nm Titanium und 80 nm Platinum are visible.

3.2 Membrane properties

The poling of the test samples was carried out at 100°C with a voltage of 12.5 kV/mm for 30 min. The curvature and centre deflection of the membranes is measured using a white-light interferometer.

For comparison the deflection vs. the actuation voltage is calculated in a FEA model assuming a transverse piezoelectric coefficient e_{31} of approx. e_{31} = -11 C/m^2. This value showed the best fit as compared to the experimentally determined membrane deflection.

Directly after the preparation the membranes of the test samples showed an initial curvature up to 0.5 -1 µm which is very likely stress induced. Obviously the sputter process of the PZT film generates mechanical stress in the PZT layer in the range of approx. 40 MPa. Besides this pre-deflection of the membranes the tests showed minor variations in their responds on the actuation voltage and also a non-linearity in the voltage dependency of the

deflections. The variability of the samples prepared is attributed to minor variations in the actual material composition.

4. SUMMARY

Crack and void free polycrystalline PZT thin films in the range of 5 µm to 15 µm have been successfully deposited on silicon substrates using a novel high rate sputtering process. With this sputter process sputter rates of 200-250 nm/min were achieved and complete 6” wafers have been covered with a 12µm thick PZT film within 60 min.

The PZT layers show a high dielectric constant ε_r between 1000 and 1800, a distinct ferroelectric hysteresis with a remanent polarisation of 17 µC/cm^2 and a coercive field strength of 5.4 kV/mm. Measurements of the piezoelectric coefficient $d_{33,f}$ have shown values of the reverse effect up to 79,7 pm/V.

Based on this new deposition process a simple membrane actuator consisting of a SOI and a sputtered PZT layer was prepared. The deflection of this membrane actuator depending on the driving voltage was measured with a white light interferometer and compared to the results of a FEA model. With this approach a transverse piezoelectric coefficient e_{31} = -11 C/m^2 was approximated.

Still in an early stage of work the Gas Flow Sputtering approach for high rate deposition of PZT thin films has shown its high potential for application in MEMS actuators. Work is ongoing for optimisation and stabilisation of the PZT deposition process and to increase the piezoelectric coefficient $d_{33,f}$ and $d_{31,f}$ respectively. Additionally the process development is extended to process 8" wafers and more methods are developed for PZT film structuring.

REFERENCES

[1] A. C. Jones, et al., “MOCVD of Zirconia and Lead Zirconate Titanate Using a Novel Zirconium Precursor”, J. Eur. Cer. Soc. 19, p. 1434, 1999.

[2] P. Muralt, et al., “Fabrication and characterisation of PZT thin-film vibrations for micromotors”, Sensors and Actuators A 48, p. 157, 1995.

[3] H. Kueppers, et al., “PZT thin films for piezoelectric microactuator applications”, Sensors and Actuators A 97-98, p. 680, 2002.

[4] M. Koch et al., “A novel micropump design with thick-film piezoelectric actuation”, Meas. Sci. Technol. 8, p. 49-57, 1997.

[5] H. Jacobsen et al., "Development of a piezoelectric lead titanate thin film process on silicon substrates by high rate gas flow sputtering", Sensors and Actuators A, 133, p. 250, 2007.

[6] K. Ishii, "High-rate low kinetic energy gas-flow-sputtering system", J. Vac. Sci. Technol. A 7, p. 256–258, 1989

[7] H. Jacobsen, K. Prume, H.-J- Quenzer, B. Wagner, K. Ortner, Th. Jung "High-rate sputtering of thick PZT thin films for MEMS", J. Electroceramics, Springer, in press.

[8] K. Prume, P. Muralt, F. Calame, Th. Schmitz-Kempen, S. Tiedke, "Piezoelectric thin films: Evaluation of electrical and electromechanical characteristics for MEMS devices", IEEE Trans. UFFC., 54, p. 8, 2007.

[9] J. Baborowski, "Microfabrication of piezoelectric MEMS, J. Electroceramics", 12, p. 33, (2004).

[10] T. Maeder, L. Sagalowicz, P. Muralt, "Stabilized Platinum Electrodes for Ferroelectric Film Deposition using Ti, Ta and Zr Adhesion Layers", Jpn. J. Appl. Phys., 37, p. 2007, 1998.