Preparation of functional PZT Films on 6'' and 8'' Silicon Wafers by High Rate Sputtering

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ABSTRACT

Crack and void free polycrystalline Lead Zirconate Titanate (PZT) thin films in the range of 2 μm to 8 μm have been successfully deposited on silicon substrates using an improved set-up for a novel high rate sputtering process. An optimised, rectangular cathode geometry (width 250 mm) allows homogenous PZT deposition on temperature controlled wafers up to 200mm diameter. Deposition rates of 100nm/min to 120nm/min for uniform layers of high quality were achieved. The sputtered PZT layers show a high dielectric constant ε_r up to 2300 and a distinct ferroelectric hysteresis loop with a remnant polarisation of 17 $\mu C/cm^2$ and a coercive field strength of 5.4 kV/mm. Piezoelectric coefficients $d_{33,f}$ of 102 to 108 pm/V have been determined using a standard aixACCT. A transverse piezoelectric module e_{31} = -10.9 C/m^2 was measured.

Experiments for structuring of deposited PZT films have started. We report results from wet etch and dry etch tests using an APS tool from the company STS.

Keywords: MEMS, actuator, PZT, gas flow sputtering

1 INTRODUCTION

Piezoelectric materials are promising candidates for powerful actuators in MEMS devices [1]. 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 lower voltage operation and low energy consumption. In order to design large force MEMS actuators required for instance in microfluidic applications 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 [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. Screen printing methods [6] suffer from high firing temperatures above 900°C.

In the following results are presented for an improved version of a new high rate sputter process for the deposition of high quality PZT films with a thickness of 2μm to 8μm. Gas flow sputter technology and initial experiments for PZT preparation by a gas flow process have been reported [7]. For further production of MEMS functional devices from the deposited film material structuring technologies will have to be applied. First results from dry and wet etch processing are presented.

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 the improved set-up used in this work a rectangular sputter source with dimensions 250 mm x 40mm was used. The substrate holder was moved linearly in front of the sputter source to deposit a uniform film on silicon wafers of up to 8'' (200 mm) diameter.

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 [8, 9]. Deposition rates of 100-120nm/min have been achieved for films of high quality.

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 immiscibility making target manufacturing of the desired material composition 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 stripes of lead and an alloy of 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 600 °C during sputtering. In order to obtain the desired crystallographic microstructure of the PZT films, the deposition temperature is of major importance. In the deposition process described here temperature can be varied independently from the process parameters of the sputter source. Therefore temperature adjustment is used as a further, independent parameter to adjust the desired material properties. Experiments have shown that the close proximity of the cool sputter cathode in front of the heated wafer under vacuum conditions acts as an efficient radiation cooling sink for the wafer surface even at constant chuck temperature. Therefore additional IR radiation heaters in front of the wafer surface beside the opening of the sputter source had to be installed to stabilise wafer surface temperature during the whole deposition process. The effect of additional IR-heaters during a deposition run is shown in Fig. 2. Experiments showed that a minimum temperature above 450ºC is necessary to initiate a crystalline growth of the PZT film, while temperatures of up to 600°C are required for the deposition of PZT films with good piezoelectric properties.
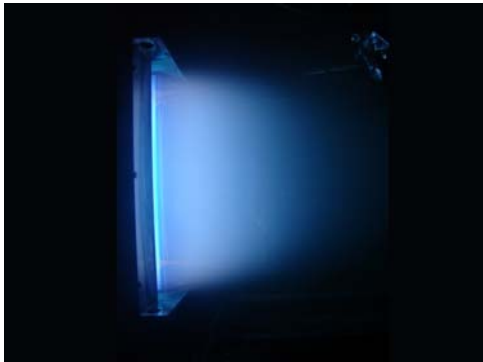

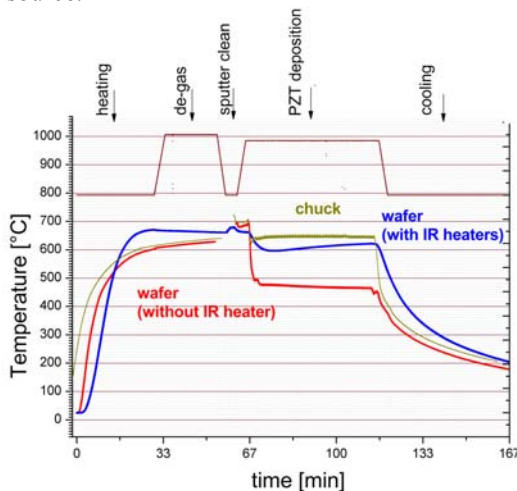Figure 1: Glow of gas discharge at the exit from rectangular sputter source.

Figure 2: Wafer surface temperature during wafer transport in front of sputter source during deposition process.

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 is 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 microanalysis (EPMA). Material composition in the sputter source was adjusted for the desired material quality of sputtered films. The stable sputter process leads to an excellent reproducibility of the films produced. The new results confirm earlier observations that the linear sputter source guarantees a very good process stability and reproducibility especially with respect to the chemical compositions of the PZT films.

3   RESULTS

Thickness of PZT films was determined by SEM (Fig. 3, 4). Deposition rates of 100-120 nm/min were adjusted with the sputter source. These high sputter rates are about 3 - 6 times higher than reported from reactive magnetron sputtering [3] and demonstrate the potential of this sputtering technique. Typical layers have a thickness of 4 µm sputtered in about 30 min. However, even 8 µm thick PZT films were deposited without cracking or delamination within 90 min.

Variation of wafer temperature during PZT deposition influences the growth process of microcystals on the wafer surface leading to different film morphologies while the material stochiometry remains identical. This effect can be utilised for optimisation of various piezoelectric material parameters. In figures 3. and 4. the influence of two different deposition temperatures on the crystal structure can be compared.
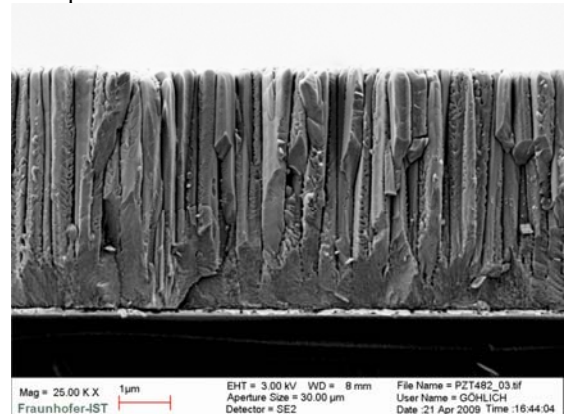

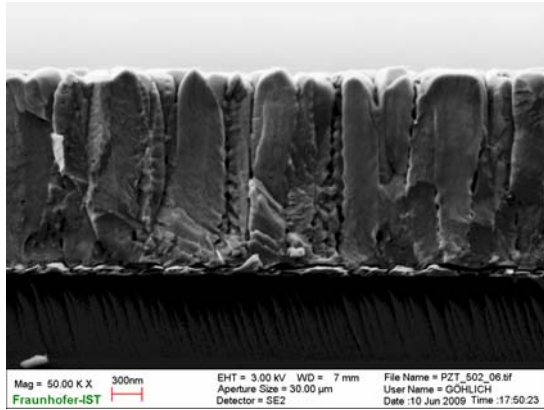Figure 3: SEM cross section of a 4.6µm thick PZT film, deposited at a wafer temperature of 480ºC.

Figure 4: SEM cross section of a 2µm thick PZT film, deposited at a wafer temperature of 580ºC. Note, SEM magnification is two times as compared to figure 3.

Poling of the test samples was carried out at 150º C with a voltage of 15kV/mm for 10 min.

Electrical measurements of the relative permittivity ε_r show a typical value of 2000, in some samples even values of 2300 have been measured. A distinct ferroelectric hysteresis loop with a remnant 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 standard aixACCT double-beam interferometer [10] show values of the reverse effect of 102 - 108 pm/V. From the measured displacements at the specific geometries of 2µm PZT on silicon test samples a corresponding $e_{31,f}$ value of -10.9C/m^2 was determined.
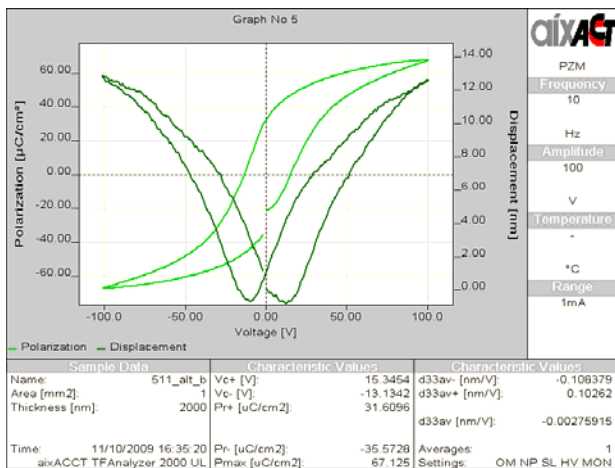

Figure 5: Result of $d_{33,f}$ measurement of 2µm thick PZT samples sputtered on a silicon substrate.

3.1 Pattern transfer in PZT films

To integrate sensing or actuating structures into a MEMS device in many cases the PZT films must be patterned or 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 is used [11].

Tests ended up with a wet 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 for 5µm thick PZT films. Due to the HF content and the required etching time the photo resist shows strong delamination effects leading to a poor quality in transferring the pattern into the PZT layer.

Thus in further experiments 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 involatile 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. Therefore the maximum sputter angle is limited to approx. 70º.
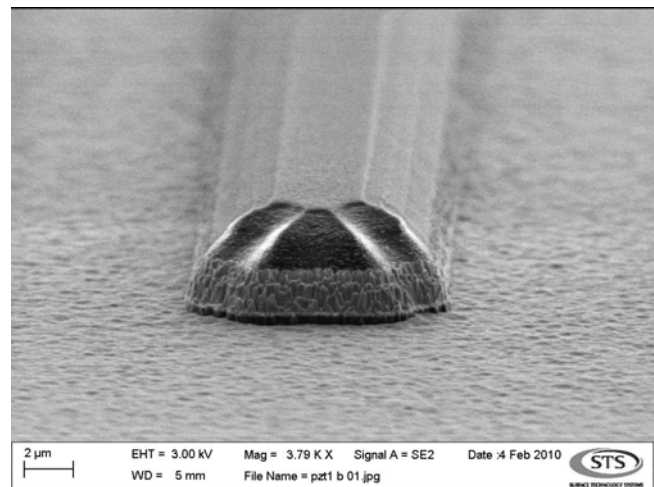

Figure 6: SEM cross section of a 2µm thick PZT layer etched by dry etching using a photo resist mask (on top).

A dry etch rate for PZT of approx. 275nm/min was observed with a selectivity over resist of 2 to 3.5:1. The dry etch process shows a very low selectivity between PZT and Pt which is used as the bottom electrode of the PZT layer [12]. In figure 6 the Pt bottom electrode has been removed,

too. In further experiments the etch process could be adjusted not to damage the bottom electrode. A uniformity of the etch process of approx. 4% over an 8" wafer can be achieved.

4. SUMMARY

Crack and void free polycrystalline PZT thin films in the range of 2 μm to 8 μm have been successfully deposited on 8" silicon substrates using a novel high rate sputtering process. With this sputter process sputter rates of 100-120 nm/min were achieved and complete 8" wafers have been covered with up to 8μm thick PZT films within 90 min.

A stable PZT deposition process can be adjusted with excellent reproducibility of the film material parameters.

The PZT layers show a high dielectric constant ε_r of up to 2300, a distinct ferroelectric hysteresis with a remnant 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 of 102 to 108 pm/V. From test samples an $e_{31,f}$ value of -10.9C/m² can be determined.

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. With dry etching a maximum sputter angle of 70º can be achieved.

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 industrial production of MEMS devices. 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.

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