Thickness mode high frequency MEMS piezoelectric micro ultrasound transducers

F. Dauchy · R. A. Dorey

Received: 22 May 2006 / Accepted: 21 August 2007 / Published online: 13 September 2007 © Springer Science + Business Media, LLC 2007

Abstract Thickness mode piezoelectric micro-electromechanical system (MEMS) ultrasound transducers, operating in the 50-75 MHz range, have been fabricated using a composite sol gel technique in combination with wet etching. The composite sol gel technique involves producing a PZT powder/sol composite slurry, which when spun down yields films a few micrometers thick. Repeated layering, and infiltration, has been used to produce PZT films between 20 and 40 µm thick. Due to the low firing temperature (<720°C) it has also been possible to integrate these PZT films with a micro-machined silicon support wafer. These PZT thick films have been structured using a wet etching technique to create free standing pillars that have been shown to resonate in thickness mode in the frequency range of 50-75 MHz. Examples of these structures and their resonant behaviour are presented.

Keywords $PZT \cdot Thick Film \cdot Ultrasound \cdot Transducer \cdot pMUT$

1 Introduction

High frequency medical ultrasound transducers operating in the region of 30–70 MHz offer the potential for high resolution imaging for ophthalmology, dermatology and intravascular applications [1, 2].

Existing medical and non distributive testing (NDT) piezoelectric transducers operate at lower frequencies (ca 1-10 MHz) and have been fabricated using diced, filled and

lapped bulk piezoelectric materials [1, 3] operating in thickness mode or micromachined silicon/piezoelectric diaphragm structures operating in bending mode [4]. The challenge faced with producing high frequency transducers is that as the resonant frequency of the transducers increases it becomes necessary to reduce the thickness/ element size of the thickness mode transducers [1] or decrease the size of the diaphragm structures. Therefore, the realisation of high frequency piezoelectric transducers represents a significant technical challenge as both diaphragm and thickness mode type transducers become increasingly difficult to manufacture. One potential route to fabricate such high frequency transducers is to integrate the piezoelectric material with a micromachined system to produce a thickness mode piezoelectric micro ultrasound transducer (Tm pMUT). For such a system to resonate at between 30 and 70 MHz requires a piezoelectric ceramic structure between 20 and 40 µm thick [1]. The production of films of such thickness using techniques such as screen printing is possible, however, issues associated with material compatibility (especially between lead containing piezoelectrics such as lead zirconate titanate (PZT) and silicon) become an issue with the required processing temperatures [5, 6].

This work presents a thick film deposition and patterning technique that can be used to produce resonant piezoelectric structures, with interconnected electrodes, on a silicon substrate.

2 Experimental

The substrates used in this work were (100) silicon wafers with a 400 nm thermal oxide (SiO_2) and 40 nm ZrO₂ layer on top. Platinum bottom electrodes, 100 nm thick, were

F. Dauchy (⊠) · R. A. Dorey Nanotechnology Group, Cranfield University, Cranfield, Bedfordshire, UK e-mail: r.a.dorey@cranfield.ac.uk

deposited using a lift off process and following DC sputtering.

The thick PZT film was deposited using a composite sol gel route [7] whereby a 2ME based sol was mixed with PZT powder of comparable composition to produce an ink. This ink was spun onto the substrate at 2,000 rpm, dried at 200°C and pyrolysed at 450°C to produce an amorphous ceramic structure. Two such layers were deposited prior to increasing the density of the film by infiltrating the structure with pure sol, spinning, drying and pyrolysing as before. This infiltration process was repeated four times per two composite layer structure. The thickness of the film was controlled by varying the number of composite layers deposited. Sixteen infiltrated composite layers were deposited to build up a film 24 μ m thick.

Once the film had been deposited onto the silicon substrate a photoresit (AZ4562) was deposited over the film, exposed and developed to produce an etch mask. The PZT film was then etched using a HF/HCl etchant [HF (0.5 vol.%), HCl (4.5 vol.%), H₂O (95 vol.%)]. Following etching a white residue was observed on the surface of the PZT. Previous work by Zheng [8] had identified this as PbClF formed as a result of the etching process. The PbClF was removed using an ultrasound water wash at the end of the etching process.

Following etching a platinum top electrode was depositing using a lift off/sputtering process. With the device fully constructed the PZT was crystallised using a thermal treatment at 720°C for 20 min in a conventional chamber furnace using a ramp rate 60°C/min and furnace cooling.

Following crystallisation of the PZT material, the silicon was removed directly below the resonant structure using TMAH wet etching. A schematic of the final device structure is shown in Fig. 1.

The final device was poled using contact poling where the sample was heated to 130°C and a field of 8 V/ μ m was applied for 5 min. The sample was the cooled to below 100°C before the field was removed. Measurement of resonant behaviour was conducted using a 8753D Hewlett Packard VNA (Vector Network Analyser) with flexible Kconnector cables. Calibration was carried out with an impedance standard substrate (ISS101-190) and SOLT (Short, Open, Load, Through) method.



Fig. 1 Schematic of Tm-pMUT device



Fig. 2 Surface of PZT film after sintering and coating with Ti/Pt showing a low level of river bed cracking and the ability to apply an uninterrupted top electrode

3 Results

3.1 PZT thick film deposition

The films produced in this work show a very low level of 'riverbed' cracking (Fig. 2). Riverbed cracking is characterised by vertical cracks which split the surface of the film into smaller cells. While a degree of such cracking can be tolerated as it does not significantly affect the 33 piezoelectric properties of the film, excessive cracking can lead to areas of the device becoming electrically and mechanically isolated with a corresponding decrease in performance. Such cracking arises when the ceramic particles within the ink are no longer able to move to accommodate shrinkage caused by the removal of the carrier fluid. This causes the internal tensile stress to increase until the bonding between individual particles is disrupted. The low amount of



Fig. 3 Fracture cross-section of PZT thick film on silicon showing the homogenous through-thickness structure



Fig. 4 Plan view of etch wall profile showing a comparison 12[C+4S] (*left*) and 6[2C+4S] (*right*) thick films showing how the inhomogeneous through thickness microstructure of the 12[C+4S] system results in a stepped etch wall profile

cracking observed in these films indicates that a controlled drying process has been achieved such that particle rearrangement is possible.

Along with the low level of riverbed cracking the deposition process also results in the formation of a very uniform through-thickness microstructure (Fig. 3). This becomes more obvious when the etch side walls of a C+4S and 2C+4S system are compared (Fig. 4). The use of the 2C+4S layering technique results in a smooth side wall profile as would be expected for an isotropic etch profile. The C+4S system exhibits a marked stepped structure which is caused by the inhomogeneous through thickness microstructure and associated different etch rates. In this instance horizontal etching is faster than vertical etching which results in the distinctive stepped pattern.

3.2 PZT wet etching process

As can be seen in Fig. 5 it is possible to structure the PZT thick films using a wet chemical etch technique. The etch times for different thicknesses of PZT are presented in



Fig. 5 Scanning electron micrograph showing the bottom electrode and patterned PZT structure of the thickness mode piezoelectric micromachined ultrasound transducer

 Table 1 Comparison of etch rates between two thicknesses of PZT film.

2C+2S units	Film thickness	Time to etch	Etch rate
5	22	5 min 18 s	4.2 μm/min
7	26	6 min 30 s	4.0 μm/min

Table 1. The decrease in etch rate with increasing thickness observed is a results of the formation of the PbClF reaction product. As this reaction product does not form an impervious layer it is still possible for the etching process to continue, albeit at a reduced rate. The PbClF reaction product was not removed at regular intervals during the etch process as the removal process would also have removed the protective photoresit etch mask.

3.3 Device characterisation

The device response shown in Fig. 6 demonstrates a resonance at approximately 70 MHz in air. Work is currently underway to compare the results obtained with those predicted using an analytical model in order to elucidate the material properties of the PZT thick material. Initial results from a range of samples with different levels of porosity show a thickness mode coupling coefficient (k_1) that can be varied between 0.2 and 0.4 with higher values obtained for higher density systems.

3.4 Conclusions

It has been demonstrated that a low temperature composite sol gel route can be used to produce patterned lead zirconate titanate (PZT) micro-features on silicon substrates. Such structures, incorporated with integrated electrodes and micromachined silicon, can be used as piezoelectric micro ultrasound transducers operating at frequencies above 50 MHz. Characterisation of such single element devices has show the structures to resonate and exhibit a value of k_t between 0.2 and 0.4 depending on the density and structure of the PZT material.



Fig. 6 Response for a 24-µm thick device structure

Acknowledgements The work presented here has been funded through a EC framework 6 project (MUSTWIN project: NMP2-CT-2003-505630) and the Royal Academy of Engineering / Engineering Physical Science Research Council UK.

References

- A. Abrar, D. Zhang, B. Su, T.W. Button, K.J. Kirk, S. Cochran, Ultrasonics, 42, 479 (2004)
- 2. W.W. Wolny, J. Euro. Ceram. Soc, 25, 1971 (2005)

- R.E. Newnham, L.J. Bowen, K.A. Klicker, L.E. Cross, Materials & Design, 2, 93 (1980). P. Muralt, J. Baborowski, J. Electroceramics 12, 101 (2004)
- F. Akasheh, T. Myers, J.D. Fraser, S. Bose, A. Bandyopadhyay, Sensors and Actuators A, 111, 275 (2004)
- 5. R.A. Dorey, R.W. Whatmore, J. Electroceramics 12, 12 (2004)
- M. Hrovat, J. Holc, S. Drnovšek, D. Belavič, J.Cilenšek, M. Kosec, J. Euro. Ceram. Soc. 26, 897 (2006)
- 7. R.A. Dorey, S.B. Stringfellow, R.W. Whatmore, J. Euro. Ceram. Soc, 22, 2921 (2002)
- K. Zheng, J. Lu, J. Chu, Microprocesses and Nanotechnology Conference International 2003. Digest of Papers, 29–31, 248 (2003)