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# Thickness mode high frequency MEMS piezoelectric micro ultrasound transducers

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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 40µm thick. Due to the low firing temperature (<720ºC) 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, Thick Film, Ultrasound, Transducer, pMUT

## 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].

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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.

**Experimental**

The substrates used in this work were (100) silicon wafers with a 400nm thermal oxide ($SiO_2$) and 40nm $ZrO_2$ layer on top. 100nm thick platinum bottom electrodes were 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 2000 rpm, dried at 200ºC and pyrolysed at 450ºC to produce an amorphous ceramic structure. 2 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 4 times per 2 composite layer structure. The thickness of the film was controlled by varying the number of composite layers deposited. 16 infiltrated composite layers were deposited to build up a film 24 µm 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%), $H_2O$ (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 720ºC for 20 minutes in a conventional chamber furnace using a ramp rate 60ºC/minute 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 figure 1.

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

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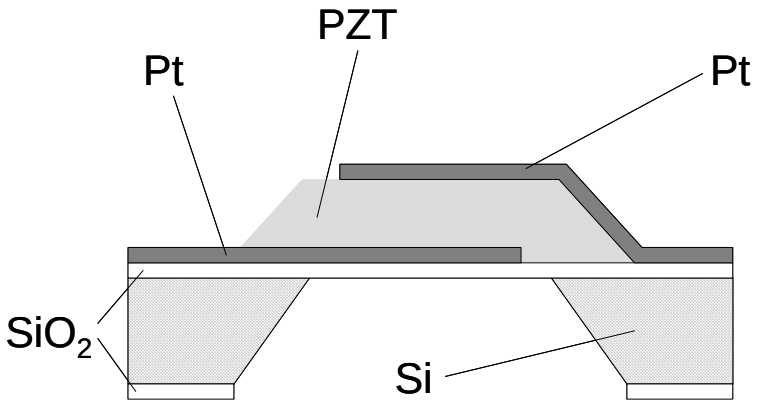


Fig. 1. Schematic of Tm-pMUT device

**Results**

*PZT thick film deposition*

The films produced in this work show a very low level of 'riverbed' cracking (figure 1). Riverbed cracking is characterises 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 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 (figure 3). This becomes more obvious when the etch side walls of a C+4S and 2C+4S system are compared (figure 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.

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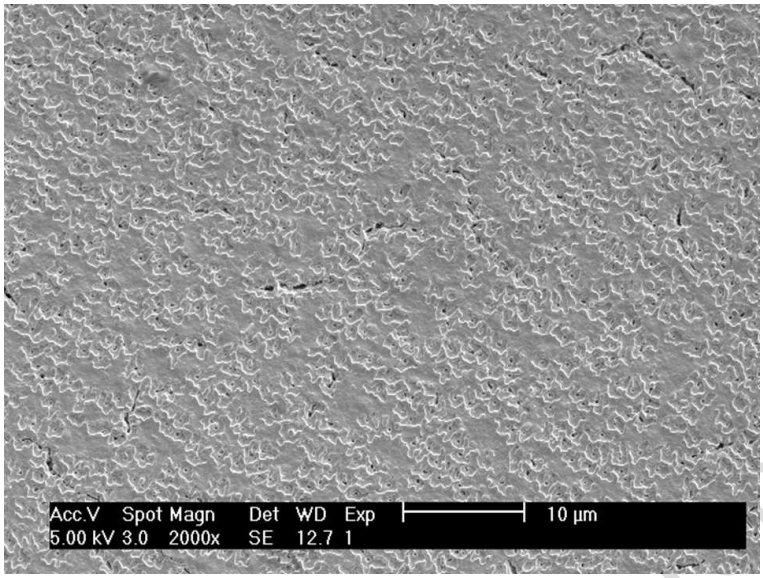


Fig. 2. Surface of PZT film after sintering and coating with Ti/Pt showing a low level of river bed cracking and the ability to apply an uninterrupted top electrode.

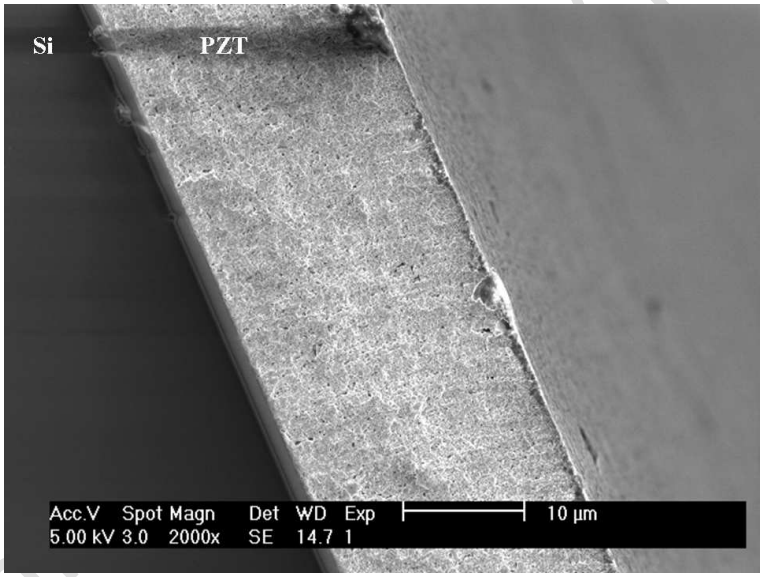


Fig. 3. Fracture cross-section of PZT thick film on silicon showing the homogenous through-thickness structure

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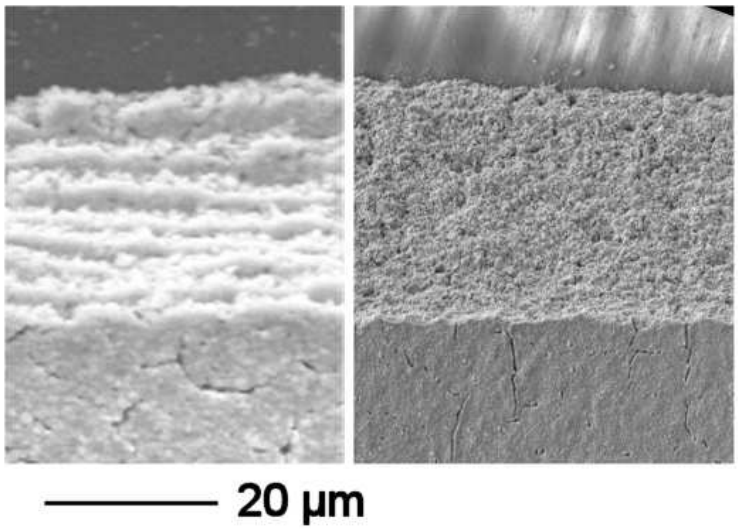


Fig. 4. Plan view of etch wall profile showing a comparison 12[C+4S] (left) and 6[2C+4S] (right) thick films showing how the inhomogeneous through thickness microstructure of the 12[C+4S] system results in a stepped etch wall profile

*PZT wet etching process*

As can be seen in figure 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 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.

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 30s | 4.0 µm/min |

*Device characterisation*

The device response shown in figure 6 demonstrates a resonance at approximately 70MHz 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 ($k_t$) that can be varied between 0.2 and 0.4 with higher values obtained for higher density systems.

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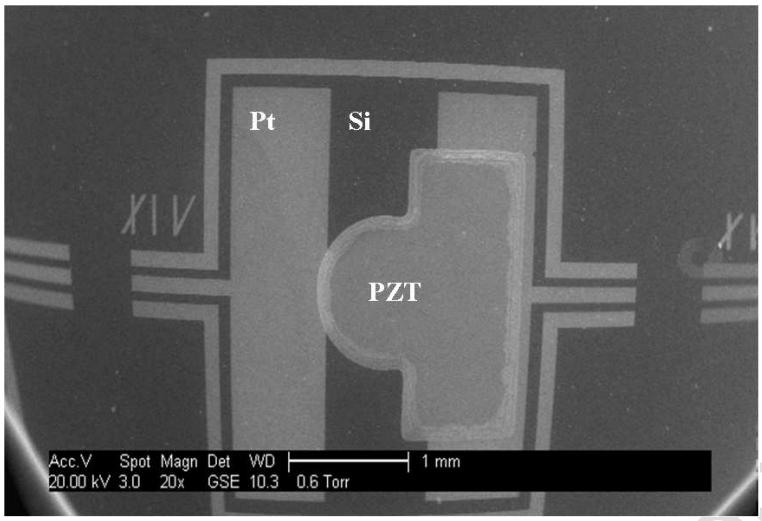


Fig. 5. Scanning electron micrograph showing the bottom electrode and patterned PZT structure of the thickness mode piezoelectric micro-machined ultrasound transducer.

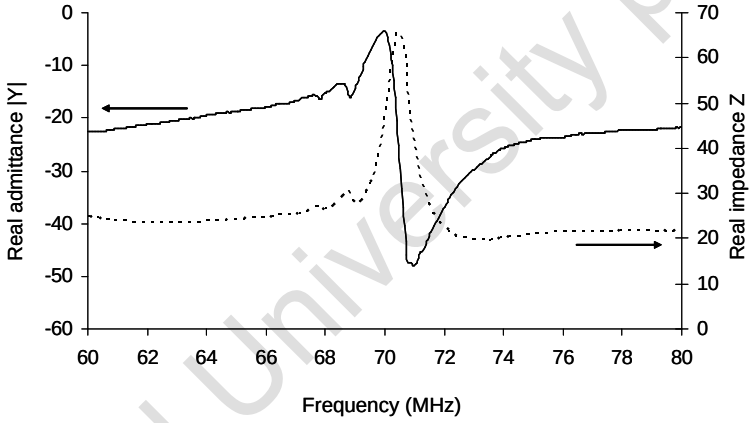


Fig. 6. Response for a 24µm thick device structure

*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 50MHz. 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.

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**References**

1. 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).
3. 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).
4. 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).
6. 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).
8. K. Zheng, J. Lu, J. Chu, Microprocesses and Nanotechnology Conference International 2003. Digest of Papers, 29-31, 248 (2003).

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