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## Micro-Machined High-Frequency (80 MHz) PZT Thick Film Linear Arrays

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### Abstract

This paper presents the development of a micro-machined high-frequency linear array using PZT piezoelectric thick films. The linear array has 32 elements with an element width of 24  $\mu\text{m}$  and an element length of 4 mm. Array elements were fabricated by deep reactive ion etching of PZT thick films, which were prepared from spin-coating of PZT sol-gel composite. Detailed fabrication processes, especially PZT thick film etching conditions and a novel transferring-and-etching method, are presented and discussed. Array designs were evaluated by simulation. Experimental measurements show that the array had a center frequency of 80 MHz and a fractional bandwidth ( $-6$  dB) of 60%. An insertion loss of  $-41$  dB and adjacent element crosstalk of  $-21$  dB were found at the center frequency.

### I. Introduction

Fabrication of high-frequency ( $\geq 30$  MHz) ultrasonic linear arrays has remained a challenge following more than a decade of study. The task is even more difficult to build arrays at a frequency greater than 80 MHz; these arrays have the potential to provide a more detailed delineation of skin anatomy for early diagnosis of cancers such as melanoma or to image small structures in other parts of the body. Recent research results have shown that micro-machining technology is a possible solution for high-frequency fabrication even though much improvement remains to be made [1]–[4]. Recently, piezoelectric films have already been widely used to fabricate micro-scale devices [5]–[8]. Among all piezoelectric films, PZT thick films have been shown to exhibit good dielectric and piezoelectric properties, making them possible ultrasonic transducer material candidates for high-frequency applications via micro-machined technology [9], [10]. Moreover, the use of PZT ceramic, PZT composite, and PZT

film for fabricating high-frequency kerfless linear arrays has been reported [11]–[13]. Although the kerfless approach is a simpler solution to fabricate linear arrays in which only the electrodes of the array elements need to be patterned onto the surface of the piezoelectric substrates [14], [15], the increased crosstalk between adjacent elements is always an issue in kerfless array. To reduce the crosstalk, mechanical isolation between elements is necessary. Ritter *et al.* reported a 30-MHz kerfed linear array by a dice-and-fill approach, where a piezoelectric plate is separated by mechanical dicing and a polymer is infiltrated and cured within the kerfs [16]. Liu *et al.* used a novel interdigital pair bonding technique to build 20- to 50-MHz linear arrays [17]. However, it is difficult to fabricate linear arrays at frequencies higher than 50 MHz with those methods because the pitch is very small.

Recently, laser machining has shown promise in applications for such high-frequency ultrasonic arrays [18], [19]. Both mechanical dicing and laser machining share the same drawback: they can only produce the isolation kerfs one by one. It is time-consuming to fabricate multi-element arrays with those methods. Microelectromechanical systems (MEMS) etching technology, however, is capable of bulk production of miniature devices. Among a wide variety of etching methods, deep reactive ion etching (DRIE) has been widely used in etching PZT thin films for ferroelectric memory applications [20]. Recently, new advances in PZT dry etching have been reported [21]–[23]. Pure sulfur hexafluoride ( $\text{SF}_6$ ) or a mixture of  $\text{SF}_6$  and Ar were used as etching gases in plasma etching of PZT. The advantages of fluorine chemistry are its good selectivity to mask materials and its relatively high etching rate, which can reach values as high as  $0.25 \mu\text{m}/\text{min}$ . However, because the components produced by PZT etching have significantly higher boiling temperatures, their removal is inefficient; in closely spaced structures, this would result in sidewall angles less than  $80^\circ$ , which is unacceptable for fabricating high-frequency arrays. On the other hand, Marks *et al.* [24] reported that the use of chlorine-based etching gases with an elevated wafer temperature yielded better vertical etch profiles and a higher etch rate. Moreover, the dry etching mechanism of PZT in chlorinated gas plasmas has been investigated in detail [25], [26], and several measures have been proposed to minimize etching damage to the PZT material during the dry etch process [27]. In this work, process parameters for the chlorinated gas plasma have been optimized and applied to PZT film etching for high-frequency array fabrication.

## II. Experimental Procedure

### A. PZT Film Preparation

The use of PZT films in high-frequency ultrasonic transducer applications requires thick, dense, and crack-free films with acceptable piezoelectric and dielectric properties. Spin-coating of solgel composite solution with PZT powder was used here to produce the PZT thick films (see Fig. 1). High quality PZT films cannot be deposited directly on silicon by the solgel process because of a thermal stress effect. Buffer layers are needed to prevent interdiffusion and oxidation reactions of the PZT film with silicon. PZT/Pt/Ti/SiO<sub>2</sub>/Si is the most widely applied sequence. Ti is an adhesion layer. Platinum (Pt) does not inhibit the diffusion of Ti to PZT side, where it reacts with oxygen and serves as nucleation centers for PZT. Because the lattice constant of the Pt is rather close to that of PZT, the PZT tends to grow in the (111) direction on Pt. Platinum-coated silicon wafers purchased from Nova Electronic Materials, Ltd. (Flower Mound, TX) were used as the substrate. The prepared composite solutions were spin-coated onto the substrates, followed by a two-step pyrolysis and a rapid thermal process (RTP). This process was repeated multiple times until the desired thickness was achieved. The optimized film fabrication procedures were described in [28] and [29]. The prepared films were found to have a thickness of  $\sim 20 \mu\text{m}$ , dielectric constant of 1250 (at 1 kHz), thickness mode electromechanical coupling coefficient  $k_t$  of 0.34 and dielectric loss of around 0.03 [13]. The density of the film was calculated to be  $6300 \text{ kg}/\text{m}^3$ , which was about 85% of bulk PZT-5H

material ( $7500 \text{ kg/m}^3$ ). The properties of PZT films and important piezoelectric materials are given in Table I and compared with the conventional piezoelectric materials. Among them, PVDF has the lowest dielectric constant, coupling coefficient, and acoustic impedance. Therefore, it has been used to fabricate high-frequency broadband single-elements transducers, but it is not suitable for miniature devices, such as array elements, because its dielectric constant is extremely low. Both PZT5H and PMN-PT have a high coupling coefficient and high dielectric constants. They are still important materials for medical transducers, even though their acoustic impedances are high. Fine-grain PZT and PMN-PT single crystal can be fabricated into high-frequency transducers as long as they don't crack during the mechanical lapping process. PMN-PT composite is a promising piezoelectric material. It exhibits excellent properties in medical transducer applications. However, fabrication cost is a big concern, and there is still not a practical solution for very-high-frequency ( $>50 \text{ MHz}$ ) PMN-PT composite transducers. PZT thick film, which is the subject of the research, shows medium-range values among the piezoelectric materials and still appears to be the most promising approach for very-high-frequency transducers up to  $500 \text{ MHz}$ .

## B. Hard Mask Deposition

The mask used to pattern the kerfed arrays is the same as the one used for the linear kerfless array [13]. The test array has 32 elements with a kerf width of  $12 \mu\text{m}$ , an element width of  $24 \mu\text{m}$ , and an element length of  $4 \text{ mm}$ . A photolithography-based method was applied to transfer design patterns onto the film surface. A layer of Cr/Au ( $500 \text{ \AA}/1000 \text{ \AA}$ ) was first patterned onto the film surface as the seed layer, then a thick positive photoresist layer was spin-coated on top of the seed layer. After baking on a hot plate, the photoresist was exposed directly by a laser writer. The wafer was dipped in photoresist developer for developing. The patterned photoresist structures were then formed. Next, the Ni electroplating process was run to form a  $4\text{-}\mu\text{m}$ -thick Ni hard mask through the openings of the photoresist pattern. To move the dense Ni layer into the small open areas, a pulse power supply (Dynatronix, Amery, WI) was used to provide pulses with  $0.1 \text{ ms}$  on and  $0.9 \text{ ms}$  off, and with an average current of  $20 \text{ mA/cm}^2$ . After electroplating, the photoresist was stripped off using acetone.

## C. Etching and Filling

The inductively coupled plasma (ICP)-reactive ion etching (RIE) dry etching technique was used to etch the film. The area underneath the nickel hard mask was protected; the rest of the area was etched off by chlorine gas plasma. The etching rate, selectivity ratio, and profile angle were the most important parameters in dry etching of PZT film for the construction of high-frequency ultrasound transducer arrays. By optimizing the ICP power, substrate power (controlling substrate DC bias voltage), and flow rates and ratios of etching gases, it was found that a median etching rate of  $8 \mu\text{m/hr}$  was a suitable value for the process. It would take too much time to achieve the final thickness at lower etch rates, whereas higher etch rates lead to deterioration of the profile angle. Finally, we used an ICP reactive ion etching system (Plasmatherm SLR770 ICP system, Unaxis Inc., Irvine, CA), which employed  $\text{Cl}_2/\text{Ar}$  based chemistry, for the etching process. At  $600 \text{ W}$  of ICP power, a DC bias voltage of  $200 \text{ V}$ ,  $20 \text{ sccm}$  of Ar, and  $60 \text{ sccm}$   $\text{Cl}_2$ , the etching rate of PZT film was  $8 \mu\text{m/hr}$ . Fig. 2 shows SEM micrographs of the etched PZT film. Results show that the thickness of the elements was  $\sim 18 \mu\text{m}$ , the sidewall angle of the elements  $> 85^\circ$ . The kerfs were infiltrated with Epo-Tek 301-2 epoxy (Epoxy Technology, Billerica, MA). Before filling, plasma cleaning was conducted to make the array surface hydrophilic, so that the epoxy penetrated more easily into the micro-scale kerfs. The degassing process was performed right after filling. The epoxy was cured at room temperature overnight followed by two hours curing at an elevated temperature of  $60^\circ \text{ C}$ . After curing, the extra epoxy and residual nickel were carefully lapped away with fine sandpapers followed by  $3\text{-}\mu\text{m}$   $\text{Al}_2\text{O}_3$  powder.

## D. Interconnection

A layer of  $\text{Si}_3\text{N}_4$  (1000 Å) was deposited as the insulating material covering the electrode area of the array before a layer of Cr/Au (500 Å/1500 Å) electrode was patterned. Figs. 3(a) and (b) shows a PZT film kerfed linear array before and after epoxy filling, respectively. The dark area in Fig. 3(b) is the region where the  $\text{Si}_3\text{N}_4$  was deposited. The backside silicon under the working area was etched with a  $\text{XeF}_2$  etching system and the  $\text{SiO}_2$  was removed by buffered oxide etch (BOE). Subsequently, conductive epoxy (E-solder 3022, Von Roll, Wädenswil, Switzerland) was filled, providing the electric connection for the backside electrode, and at the same time acting as a backing material. The 26-AWG 50-Omega; miniature coaxial cables (Cooner Wire Inc., Chatsworth, CA) were connected by conductive silver epoxy with the electrode of each element. The prototype array was housed in a copper tube. The poling process was carried out after patterning the electrodes and before gluing on wires.

## III. Results and Discussions

### A. Experimental Measurements

The measurement set-ups were the same as those used for the kerfless arrays, which were described previously [13]. A measured pulse-echo result was shown in Fig. 4 shows that after depositing a layer of parylene as a matching layer, the array had a center frequency of 80 MHz and  $-6$ -dB bandwidth of 60%. The result shows that this array of PZT films exhibited acceptable bandwidth with one matching layer.

Fig. 5 shows the uniformity of the array element performance in terms of capacitance and dielectric loss. As shown in Fig. 5, the capacitance was approximately 30 pF and the dielectric loss 0.04 except for several elements with variation from 30 to 50 pF caused by connection problems between two elements. Most importantly, there were no broken elements observed. This is a significant advantage of the micro-machined approach over the conventional dice-and-fill technology [12], [16]. Particularly, it is not possible to dice such a small kerf using our current dice saw (larger than 12  $\mu\text{m}$ ). Fig. 6 shows the measured insertion loss of the array element. After compensation for the attenuation caused by the water and reflection from the quartz target, an insertion loss of  $-41$  dB was obtained at the frequency of 90 MHz. The relatively high insertion loss was mainly due to the smaller coupling coefficient of the PZT films than a bulk ceramic. The results of combined acoustic/electrical crosstalk measurements are presented in Fig. 7. A set of results for nearest neighbor were measured. A crosstalk of around  $-21$  dB was observed between 50 MHz and 90 MHz, which was approximately 5 dB lower than the PZT film kerfless array [13] because of the kerf created between two elements.

### B. Modeling Results

Because a high-frequency array imaging system (over 50 MHz) is not yet available at our lab, both PZFlex (Weidlinger Associates Inc., New York, NY) and Field II modeling were carried out to illustrate the beamforming capability of such an array. In the simulation, eight elements were used as the active aperture. The location of the focal point was determined by the time delays and the width of the active aperture. By varying the time delays, both the transmitting and receiving beams can be focused to different points along the depth of the image. This process of dynamic focusing can dramatically improve the quality of the ultrasonic images over a fixed-focusing system. The PZFlex program was used to predict the azimuthal beam profile. The result in Fig. 8(a) demonstrates that, with dynamic focusing delays, the array could be well focused at 1 to 1.5 mm, and a transmitting grating lobe appears at approximately  $40^\circ$ . The imaging capability with the array was simulated using a Field II program with the same active aperture and time delay data. Fig. 8(b) shows the simulated image of a point scatter phantom. As shown, a better image spatial resolution was obtained at the focal zone (1 to 1.5 mm).

### C. Discussions

Although a kerfed PZT thick-film array was successfully fabricated with the described procedures, there were still several problems with the fabrication procedures that needed to be addressed. First, the array was likely to crack when the silicon substrate was removed and E-solder was filled. Second, with the method reported here, the area underneath the electrodes had to be covered with an insulating material. Therefore, a major portion of the film was wasted. Finally, it was impossible to press focus the array because of the hard silicon substrate underneath the film. Hence a modified fabrication method, as shown in Fig. 9, was introduced. In the new process, E-solder 3022 was first cast onto the front surface of the PZT thick film and centrifuged for 5 min at 3000 rpm. After curing the E-solder overnight, the PZT film was cut into  $4 \times 2$  mm pieces by a dicing saw. Then, the pieces were immersed in a KOH 20% solution at a temperature of  $80^\circ\text{C}$ . This resulted in the PZT film peeling off from the silicon substrates while still firmly bonded to the E-solder. Moreover, there was no obvious damage to the film when it was removed from the silicon substrate [29]. A plastic tube was placed concentrically around the sample. The tube was filled with Epotek-301-2 non-conductive epoxy (Epoxy Technology, Inc., Billerica, MA) and the epoxy was degassed to remove air bubbles. The film surface was sealed by Kapton tape, avoiding touching the epoxy. After curing, the extra epoxy was lapped away. After removing the silicon substrate, the flexibility of the array materials allowed the array elements to be curved in the elevational direction by press-focusing. PZFLEX simulations were used to predict the effects of the elevation focusing. In the simulation, the elevation aperture was 4 mm, and the focusing point was 4 mm as well, providing an elevation f-number of 1. Fig. 10 shows the predicted pressure field in water of the flat array and the press-focused array. The focused array produced a much narrower and more uniform beam than the non-focused one, yielding a thinner slice thickness at the focal point. The crosstalk value ( $-21$  dB) of the kerfed array is still high. The use of Epotek 301-2 as the kerf filler results in significant mechanical coupling between the elements. Better isolation could be achieved by the use of a softer and higher loss material in future work. In addition, improving piezoelectric properties of PZT films is very important to increase the sensitivity of array. With the current design, the linear array only could provide a very limited view of imaging because it had a total of 32 elements and 8 elements were used as the active aperture. This problem can be resolved by increasing the array elements ( $>256$ ) and its active aperture ( $>32$  elements). Meanwhile, a more compact layout of connection pads can be obtained by using ultrasonic wire bonding instead of the wire gluing approach. In addition, the piezoelectric properties of PZT films needs to be improved to further increase the sensitivity of the array.

### IV. Conclusions

High-frequency ( $>80$  MHz) kerfed ultrasonic linear arrays prepared from  $20\text{-}\mu\text{m}$ -thick PZT thick films were successfully fabricated using MEMS technology. Experimental results show that the array had a center frequency of 80 MHz and one element had a fractional bandwidth at  $-6$  dB of 60%. The insertion loss and crosstalk of the thick films array were  $-41$  dB and  $-21$  dB, respectively. A new fabrication process for producing focused linear arrays with more elements is on-going. The experimental results suggest that PZT thick film linear arrays may be a viable option in the development of high-frequency biomedical imaging transducer technology.

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## Biographies



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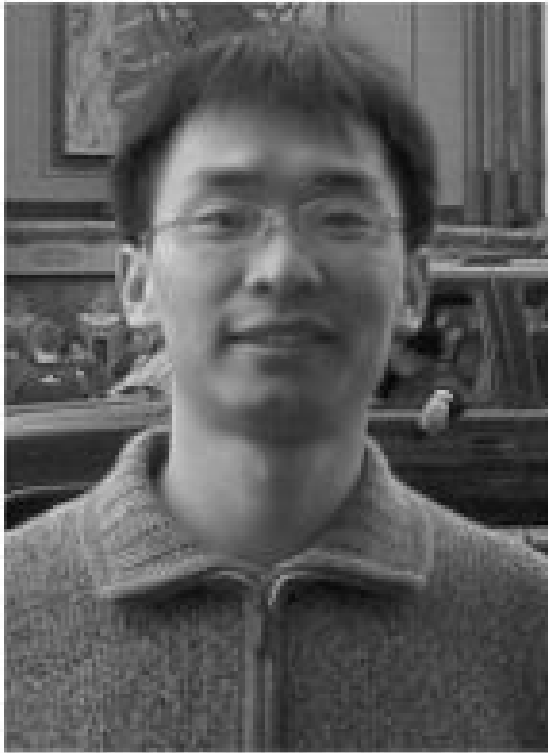
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He has been involved with microelectromechanical systems (MEMS) since 1998, and he has performed research in diverse areas within this specialty, including microfluidics, piezoelectrical microjets for drug delivery, biosensors, and micromachined high-frequency ultrasonic array transducers. His current projects include fabrication of functional piezoelectric material composite, development of high-frequency ultrasonic array transducers for medical imaging, and cell/particle manipulation in bioengineering.





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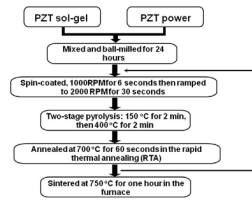
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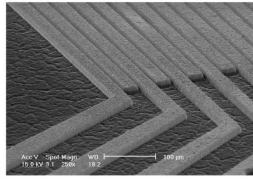
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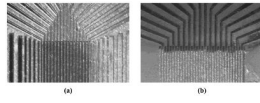
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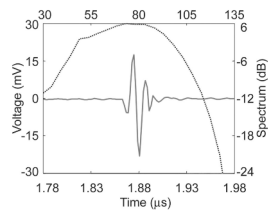
**Fig. 1.**  
The PZT thick film fabrication procedure.



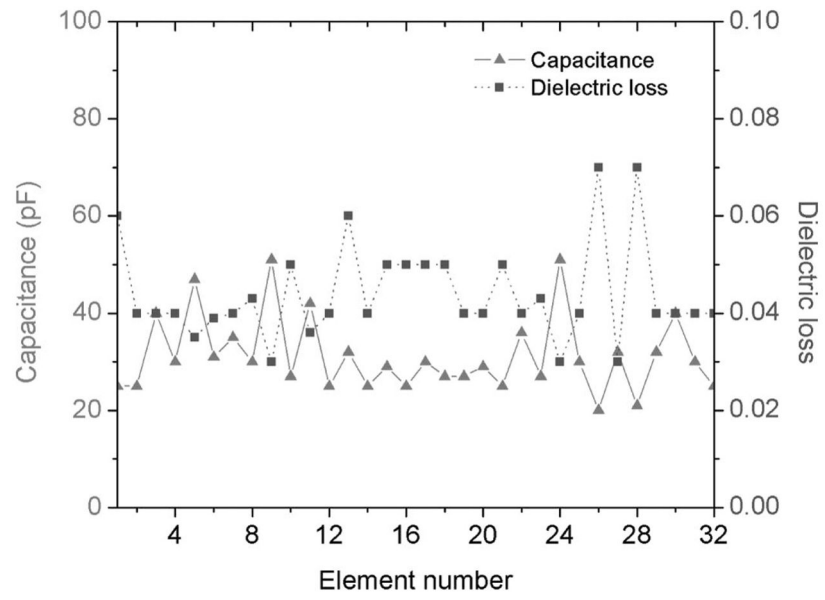
**Fig. 2.**  
Picture of a linear array dry-etched from a PZT thick film.



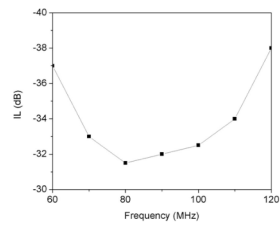
**Fig. 3.**  
Photographs of the array before and after epoxy filling.



**Fig. 4.**  
Measured pulse-echo and spectrum of the kerfed array.

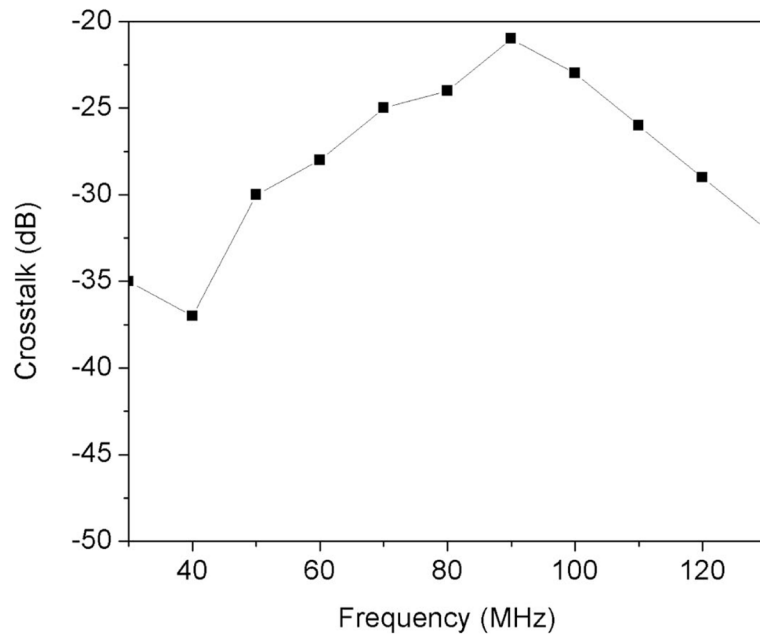


**Fig. 5.**  
Measured element uniformity of the kerfed array.

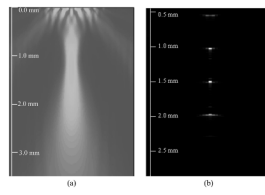


**Fig. 6.**  
Measured insertion loss of a typical kerfed array element.

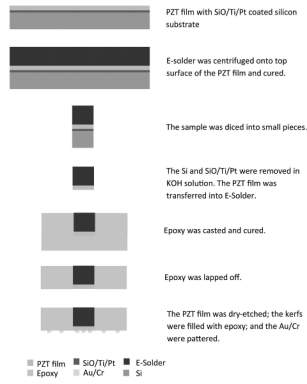




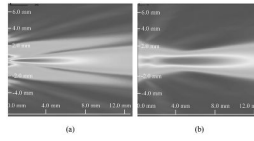
**Fig. 7.**  
Measured crosstalk between adjacent kerfed array elements.



**Fig. 8.** PZFLEX (a) and Field II (b) simulation of electrical beam focusing of the array.



**Fig. 9.** Modified fabrication flowchart of the PZT thick film linear array.



**Fig. 10.** PZFLEX simulation of pressure field in water of flat (a) and focused (b) arrays.

TABLE I

Properties of Different Piezoelectric Materials.

	PVDF	PZT5H	PMN-PT	PMN-PT 1-3 composite	PZT film*
Dielectric constant (at 1 kHz)	10	3400	~3000	~800	1000-2000
$k_t$	0.11	0.55	0.6	0.8	0.2-0.35
Acoustic impedance (MRayl)	3.4	34	37	1.5	1.5-20

\* The PZT film data varies with fabrication conditions.