Published in final edited form as:

IEEE Trans Ultrason Ferroelectr Freq Control. 2009 October ; 56(10): 2304–2310. doi:10.1109/TUFFC.2009.1311.

Very High Frequency (Beyond 100 MHz) PZT Kerfless Linear Arrays

Da-Wei Wu,

NIH Center for Ultrasonic Transducer Technology, University of Southern California, Los Angeles, CA (daweiwu@usc.edu)

Qifa Zhou [Member, IEEE],

NIH Center for Ultrasonic Transducer Technology, University of Southern California, Los Angeles, CA

Xuechang Geng,

Blatek Inc., State College, PA

Chang-Geng Liu,

Geospace Research Inc., El Segundo, CA

Frank Djuth, and

Geospace Research Inc., El Segundo, CA

K. Kirk Shung [Fellow, IEEE]

NIH Center for Ultrasonic Transducer Technology, University of Southern California, Los Angeles, CA

Abstract

This paper presents the design, fabrication, and measurements of very high frequency kerfless linear arrays prepared from PZT film and PZT bulk material. A 12- μm PZT thick film fabricated from PZT-5H powder/solution composite and a piece of 15- μm PZT-5H sheet were used to fabricate 32-element kerfless high-frequency linear arrays with photolithography. The PZT thick film was prepared by spin-coating of PZT sol-gel composite solution. The thin PZT-5H sheet sample was prepared by lapping a PZT-5H ceramic with a precision lapping machine. The measured results of the 2 arrays were compared. The PZT film array had a center frequency of 120 MHz, a bandwidth of 60% with a parylene matching layer, and an insertion loss of 41 dB. The PZT ceramic sheet array was found to have a center frequency of 128 MHz with a poorer bandwidth (40% with a parylene matching layer) but a better sensitivity (28 dB insertion loss).

I. INTRODUCTION

Fabrication of high-frequency (30 to 50 MHz) ultrasonic linear arrays is still a challenge. The task is even more difficult to build arrays at a frequency greater than 100 MHz, which has the potential to provide more detail skin texture for early diagnosis of melanoma or to image small objects such as stem cells. Recent research shows that micro electromechanical systems (MEMS) micro-machined technology is a possible solution to such high-frequency applications although much improvement still needs to be made [1]–[4]. Piezoelectric films have already been widely used to fabricate microscale devices [5]. Among the piezoelectric films, PZT film has been shown to exhibit good dielectric and piezoelectric properties, making it a possible candidate for ultrasonic transducer material [6], [7]. In recent years, it has been used to fabricate high-frequency single-element transducers, kerfless annular arrays, lateral mode linear arrays, and low-frequency 2-D arrays [8]–[12]. Although PZT films typically have a lower electromechanical coupling coefficient than bulk PZT, they have several advantages

over conventional PZT bulk materials in ultrasonic transducer applications. For example, the PZT film has relatively low acoustic impedances (~ 15 to 20 MRayls) compared with conventional PZT bulk material (~ 30 MRayls), which is useful in the fabrication of broad bandwidth ultrasonic transducers. In addition, if the sintering temperature of the PZT film can be further decreased in the future, the PZT film transducers and their processing circuits could be fabricated on the same silicon chip. In this paper, a thickness mode kerfless high-frequency PZT film linear array was developed as an alternative to kerfed arrays if a higher cross-talk level is acceptable, because dicing fine kerfs at present is still a very expensive endeavor. This issue has been addressed by C. Morton *et al.* [13] and C. E. M. Démoré *et al.* [14]. Performance of the kerfless array prepared from PZT films was compared with a PZT-5H ceramic sheet array fabricated from an identical array pattern.

II. FABRICATION OF PZT THICK-FILM LINEAR ARRAY

A. Preparation of PZT Thick Film

The fabrication of PZT thick films was based on a PZT-5H ceramic powder/sol-gel solution composite method, which was described in [12] and [15]. A 4-in $\langle 100 \rangle$ 500- μm thick silicon wafer (Nova Electronic Materials, Ltd., Carrollton, TX) with 5000- $\text{\AA}$ thermal oxide, 200- $\text{\AA}$ Ti, and 1000- $\text{\AA}$ Pt was used as a substrate. By using multilayer spin-coating of a composite solution and vacuum-infiltration of sol-gel solution [12], [16], crack-free, 12- μm thick PZT films were prepared. Fig. 1 shows SEM images of the film. The surface of the film was sufficiently smooth for array fabrication.

B. Characterization of PZT Thick Film

Several important parameters of the film were measured for modeling purposes. Circular Cr/Au electrodes with a diameter of 1.5 mm were first deposited by sputtering. Dielectric properties were measured using an Agilent 4294A impedance analyzer (Agilent Technologies Inc., Philadelphia, PA) and polarization-field (P-E) hysteresis properties were evaluated using a Radiant precision materials analyzer (Radiant Technologies, Albuquerque, NM). Fig. 2 shows the (a) measured ferroelectric hysteresis loop and (b) dielectric properties of the film, respectively. The dielectric loss tangent and dielectric constant were found to be ~ 0.03 and 1,200 at 1 kHz, respectively. The clamped dielectric constant and the effective electromechanical coupling coefficient k_t of the film were calculated to be 600 and 0.34 according to the IEEE standard on piezoelectricity [17]. This k_t value is smaller than the coupling coefficient of PZT bulk materials (0.4–0.6). The relatively low acoustic impedance, however, made the films easier to match acoustically: to achieve a bandwidth wider than 50%, 2 matching layers will be required for a conventional PZT transducer, but only one matching layer is required for a PZT thick film transducer. The density of the film was also measured. The PZT film was first cut into 10×10 -mm squares by a dicing saw. The thickness of the film (over 10 μm) was determined using a SEM (Hitachi, S-3500N, Tokyo, Japan) to allow the determination of the volume. Then, the parts were immersed in a KOH 20% solution at a temperature of 80°C. This resulted in the PZT film peeling off from the silicon substrates in approximately 10 min. The SEM images (Fig. 3) confirm that there is no obvious damage to the film when it was removed from the silicon substrate. Finally, the mass of the film was measured using a precision electronic balance (OHAUS Corporation, Pine Brook, NJ) after clearing Pt/Ti/SiO₂ debris and drying. The density of the film was calculated to be 6300 kg/m³, which is about 85% of bulk PZT-5H material (7500 kg/m³). The measured properties are summarized in Table I. The results were later used as the modeling input in PiezoCAD (Sonic Concepts, Wood-inville, WA) software.

C. Patterning the PZT Film Arrays

A mask was designed for patterning the 32-element linear arrays. In the initial design, the linear array has a kerf of 12 μm , an element width of 24 μm , and an element length of 4 mm as shown in Fig. 4.

The details of the fabrication processing are illustrated in Fig. 5. First, an insulating layer of 1- μm Si_3N_4 was deposited onto PZT thick film and patterned to leave an opening for the working area of the linear arrays. Then a layer of Au/Cr was deposited on the top surface and patterned as the top electrodes with photolithography. If the Au/Cr electrode is too thick, it will damp the center frequency. Connection will be a problem if the Au/Cr layer is too thin. Based on our experience, 1000 $\text{\AA}$ /500 $\text{\AA}$ of Au/Cr is a reasonable thickness: it can provide good conduction and only damp ~ 1 MHz of the center frequency. The backside silicon under the working area was etched with a XeF_2 etching system. Subsequently, the layer of SiO_2 was removed by BOE. Finally a conductive epoxy, E-solder 3022 (Von Roll Isola Inc., New Haven, CT), was used as filler and provided the electric connection for the backside electrode, and at the same time, acted as a backing material. Fig. 6 shows the pictures of the device fabricated. Only in the working area (marked in green), are the top electrodes directly in contact with PZT thick film. All other areas are covered by the insulating layer of Si_3N_4 .

D. Poling the PZT Film Arrays

The PZT thick film has to be properly polarized to obtain the pulse-echo signal from the array. Wang *et al.* [18] and Takahiro *et al.* [19] reported and discussed the poling conditions of PZT ceramics. But those poling conditions are not suitable for the PZT films. The poling electric field of PZT films should be much higher than that of PZT ceramics (normally $\sim 2\text{--}3$ V/ μm) [20]. A set of experiments was performed to determine the optimal poling conditions of the PZT composite films. As shown in Fig. 7, under constant temperatures, the magnitude of the output voltages of the poled transducer increased with increasing poling fields and reached a nearly constant value at around 12 V/ μm . When the poling fields were kept constant, the results suggested that the optimal poling temperature to reach maximum output voltage was around 160°C.

III. FABRICATION OF PZT SHEET LINEAR ARRAY

The powder used to fabricate the PZT thick film is PZT-5H. So PZT-5H bulk sheet was chosen to fabricate the bulk material array. The fabrication procedure is very similar to the PZT thick-film linear array. The only major difference is that PZT-5H sheet was used as the piezo-material in this experiment. A piece of bulk PZT-5H sheet was bonded onto a silicon substrate with E-solder 3022 and was later lapped to 15- μm thickness with a precision lapping machine (DA G 810 Grinder, Disco Corp., Tokyo, Japan). The same mask was used to pattern the kerfless arrays with the same microfabrication procedures described previously.

IV. RESULTS

With data listed in Table I, PiezoCAD software was used to predict the performance of the kerfless arrays. In the models, E-solder 3022 (5.9 MRayls) was selected as the backing material. No matching was added. The modeled results predicted that the center frequencies of the PZT film and PZT-5H array are 118 MHz and 132 MHz, as shown in Figs. 8 (a) and (b), respectively. The modeled results also suggested that, without any front matching, the bandwidth of the PZT film array might reach 47%, which is 17% higher than PZT-5H array (30%). After proper poling and packaging, the pulse-echo signals of the PZT film and PZT-5H arrays were successfully recorded and plotted in Figs. 9 (a) and (b), respectively. The measured pulse-echo of PZT film array is in good agreement with the modeled data. This confirms that the measured

PZT film properties in Table I are accurate. The PZT-5H array's echo shows fewer rings than the modeling result. Most likely, it was caused by either the deviation of data values provided in the PZT-5H datasheet or the fabrication errors such as the acoustic impedance of the E-solder is higher than the experimental value of the 5.9 MRays. To further improve the bandwidth of the arrays, a layer of 5- μ m parylene was deposited onto the front-surface of the arrays as the matching material. With the parylene matching, the bandwidth of the PZT film array might be as high as 60% and the bandwidth of the PZT-5H array increased to 40%. Higher bandwidth of the PZT film array is attributed to its lower acoustic impedance compared with PZT-5H sheet array. To compare the sensitivity of the arrays, 2-way insertion loss testing was performed. In the setup, a piece of 2-inch quartz was immersed in a degas water bath at room temperature as a target. 20-cycle 5-V sinusoid high-frequency bursts was generated by Tektronix AFG2020 function generator (Tektronix, Inc., Richardson, TX) as the signal sources. The echo signals were acquired and displayed using the LeCroy LC5341-GHz digital oscilloscope (LeCroy, Chestnut Ridge, NY). Because the echo signal of the thick film array is weak, the echo was first amplified by a 35-dB low-noise high-frequency Miteq AU-1466 preamplifier (Miteq, Hauppauge, NY) before feeding to the oscilloscope. Losses due to attenuation in the water bath and the transmission of the signal in the quartz target were compensated for, but the losses caused by diffraction were not included. The PZT film array and PZT-5H array was found to have insertion losses of 41 dB and 28 dB, respectively. All the above measured results are summarized in Table II. The results showed that the PZT film kerfless array had a center frequency of 120 MHz, a bandwidth of 60% with a parylene matching layer, and insertion loss of 41 dB. The PZT sheet kerfless array was found to have a center frequency of 128 MHz. The measured results revealed that the PZT sheet array has a poor bandwidth (40% with a parylene matching layer) but a better sensitivity (28 dB insertion loss).

V. CONCLUSION

High-frequency (> 120 MHz) kerfless ultrasonic linear arrays were successfully fabricated with PZT film and PZT-5H sheet. The experimental results suggest that PZT film arrays may be useful in broad bandwidth applications because of its relatively low acoustic impedances (15 to 20 MRays) compared with PZT sheet materials (~30 MRays). PZT 5H sheet, on the other hand, has greater sensitivity, because of its much higher coupling coefficient of the PZT-5H ($k_t = 0.55$) than the PZT film's ($k_t = 0.34$). The insertion loss of the PZT-5H array is 13 dB lower than PZT film array's. There is still room for further improvement of PZT film functional properties making them comparable to PZT sheet by optimization of spin-coating procedure (such as spin-coating speeds and durations), sintering condition, PZT pure solution vacuum-filling procedure (such as vacuum pressure and duration and filling sequence), and PZT pure solution to PZT powder mass ratio. Doping of PZT solution also has been shown to enhance properties of the films [21].

The next step for this work is to separate the arrays' elements with deep reactive ion etching (DRIE) dry etching technology. This will decrease the cross-talks between elements.

Acknowledgments

The authors would like to thank Benpeng Zhu and Jay Williams for their assistance with the film and transducer fabrication process.

This work was supported by NIH Grant #P41-EB2182 and NIH/NCI R44-CA110214.

References

1. Bernstein JJ, Finberg SL, Houston K, Niles LC, Chen HD, Cross LE, Li KK, Udayakumar K. Micromachined high frequency ferroelectric sonar transducers. *IEEE Trans. Ultrason. Ferroelectr. Freq. Control* 1997 Sep.;vol. 44(no 5):960–969.
2. Pang G, Sayer M, Lockwood GR, Watt M. Fabrication of PZT sol gel composite ultrasonic transducers using batch fabrication micromolding. *IEEE Trans. Ultrason. Ferroelectr. Freq. Control* 2006 Sep.;vol. 53(no 9):1679–1684. [PubMed: 16964918]
3. Ladabaum I, Khuri-Yakub BT, Spoliansky D. Micromachined ultrasonic transducers: 11.4 MHz transmission in air and more. *Appl. Phys. Lett* 1996;vol. 68(no 12):7–9.
4. Dauchy F, Dorey RA. Thickness mode high frequency MEMS piezoelectric micro ultrasound transducers. *J. Electroceram* 2007;vol. 19(no 4):383–386.
5. Muralt P. Ferroelectric thin films for micro-sensors and actuators: A review. *J. Micromech. Microeng* 2000;vol. 10:136–146.
6. Xu, F.; Wolf, RA.; Yoshimura, T.; Trolier-McKinstry, S. Piezo-electric films for MEMS applications; *Proc. 11th Int. Symp. Electrets*; 2002. p. 387-396.
7. Trolier-McKinstry S, Muralt P. Thin film piezoelectric for MEMS. *J. Electroceram* 2004;vol. 12(no 1–2):7–17.
8. Lukacs M, Sayer M, Foster FS. Single element high frequency (<50 MHz) PZT sol gel composite ultrasound transducers. *IEEE Trans. Ultrason. Ferroelectr. Freq. Control* 2000 Jan.;vol. 47(no 1):148–159. [PubMed: 18238526]
9. Dorey RA, Dauchy F, Wang D, Berriet R. Fabrication and characterization of annular thickness mode piezoelectric micro ultrasonic transducers. *IEEE Trans. Ultrason. Ferroelectr. Freq. Control* 2007 Dec.;vol. 54(no 12):2462–2468. [PubMed: 18276538]
10. Mina IG, Kim H, Kim I, Park SK, Choi K, Jackson TN, Tutwiler RL, Trolier-McKinstry S. High-frequency piezoelectric MEMS ultrasound transducers. *IEEE Trans. Ultrason. Ferroelectr. Freq. Control* 2007 Dec.;vol. 54(no 12):2422–2430. [PubMed: 18276533]
11. Dausch, DE.; Castellucci, JB.; Chou, DR.; von Ramm, OT. Piezoelectric micromachined ultrasound transducer (pMUT) arrays for 3D imaging probes; *Proc. IEEE Ultrasonics Symp*; 2006. p. 934-937.
12. Zhou, QF.; Shung, KK.; Zhang, Q.; Djuth, FT. High frequency piezoelectric micromachined ultrasonic transducers for imaging applications; *Proc. SPIE*; Orlando, FL: 2006 Apr.. p. 19-20.
13. Morton, CE.; Lockwood, GR. Evaluation of kerfless linear arrays; *Proc. IEEE Ultrasonics Symp*; 2002. p. 1257-1260.
14. Demore CEM, Brown JA, Lockwood GR. Investigation of cross talk in kerfless annular arrays for high-frequency imaging. *IEEE Trans. Ultrason. Ferroelectr. Freq. Control* 2006 May;vol. 53(no 5):1046–1056. [PubMed: 16764458]
15. Zhou QF, Cannata JM, Meyer RJ, Van Tol DJ, Hughes WJ, Shung KK, Trolier-McKinstry S. Fabrication and characterization of micro tonpilz high frequency transducer derived by PZT thick films. *IEEE Trans. Ultrason. Ferroelectr. Freq. Control* 2005;vol. 52(no 3):350–357. [PubMed: 15857042]
16. Dorey RA, Stringfellow SB, Whatmore RW. Effect of sintering aid and repeated sol infiltrations on the dielectric and piezoelectric properties of a PZT composite thick film. *J. Eur. Ceram. Soc* 2002;vol. 22(no 16):2921–2926.
17. Piezoelectricity. *ANSI/IEEE Standard*; 1987. p. 176-1987.
18. Wang, HW.; Cheng, SY.; Wang, CM. Optimization of poling process for piezoelectric PZT ceramics. 6th IEEE Electronic Manufacturing Technology Symp; Nara; Apr.; Japan. 1989. p. 263-266.
19. Takahiro Y, Masako K, Norikazu S. Influence of poling conditions on the piezoelectric properties of PZT ceramics. *J. Mater. Sci. Mater. Electron* 2000;vol. 11(no 5):425–428.
20. Damjanovic D. Ferroelectric, dielectric and piezoelectric properties of ferroelectric thin films and ceramics. *Rep. Prog. Phys* 1998;vol. 61:1267–1324.
21. Chang JF, Desu SB. Effects of dopants in PZT films. *J. Mater. Res* 1994;vol. 9(no 4):955–969.

Biographies

Da-Wei Wu is a research scientist at Industrial Research Limited in New Zealand. He received his B.S. degree from Shanghai Jiao Tong University, China, in 1999; his M.S degree from the University of Miami, Coral Gables, FL, in 2004; and his Ph.D. degree from the University of Southern California, Los Angeles, CA, in 2009; all in biomedical engineering. He was a research assistant at the Edward Dauer Bioimaging Laboratory at the University of Miami from 2002 to 2004, and at the NIH Resource Center for Medical Ultrasonic Transducers at the Biomedical Engineering Department of University of Southern California from 2004 to 2009.

His research interests are medical imaging, PZT thick films, and high-frequency ultrasonic transducers and arrays.

Qifa Zhou received his Ph.D. degree from the Department of Electronic Materials and Engineering at Xi'an Jiaotong University, China in 1993. He is currently a Research Associate Professor at the NIH Resource on Medical Ultrasonic Transducer Technology and the Department of Biomedical Engineering at the University of Southern California, Los Angeles, CA. Before joining USC in 2002, he worked in the Department of Physics at Zhongshan University of China, the Department of Applied Physics at Hong Kong Polytechnic University, and the Materials Research Laboratory at The Pennsylvania State University. His current

research interests include the development of ferroelectric thin films, MEMS technology, nano-composites, and the modeling and fabrication of high-frequency ultrasound transducers and arrays for medical imaging applications. He has published more than 70 papers in this area. He is a member of the IEEE UFFC.

Xuecang Geng received a B.S. degree in physics from Xi'an Jiaotong University, Xi'an, China, in 1983, a M.S. degree in acoustics from the Institute of Acoustics, Chinese Academy of Sciences, Beijing, China, in 1989, and a Ph.D. degree in material engineering from Pennsylvania State University, University Park, PA, in 1997.

From 1983 to 1986 he was an engineer at Center of Microelectronics, Chinese Academy of Sciences, Beijing, China. This work focused on the computer-aided design of integrated circuit modeling of the integrated circuit process. From 1990 to 1993 as a research assistant at the ultrasonic lab of the Institute of Acoustics, Beijing, China, he worked on non-destructive testing of materials, acoustic wave logging, design, fabrication, and characterization of 1–3 piezocomposites for ultrasonic transducer applications. Since 1998, he has worked as a senior scientist at Blatek, Inc., State College, PA, focusing on ultrasonic transducer design for medical and industrial applications.

Chang-Geng Liu photograph and biographies not available at time of publication.

Frank T. Djuth is the president of Geospace Research, Inc., El Segundo, CA. He received his B.S. degree in physics from the University of Pittsburgh, Pittsburgh, PA, in 1973, and his M.S. and Ph.D. degrees in physics from Rice University, Houston, TX, in 1976 and 1978, respectively. Prior to joining Geospace Research, Inc. in 1991, Dr. Djuth held a position as a research scientist at The Aerospace Corporation, El Segundo, CA. Dr. Djuth is the author of more than 120 refereed journal papers in the areas of ultrasound, MEMS, materials science, medicine, atmospheric science, and radio science.

K. Kirk Shung obtained a B.S. degree in electrical engineering from Cheng-Kung University in Taiwan in 1968, an M.S. degree in electrical engineering from the University of Missouri, Columbia, in 1970, and a Ph.D. degree in electrical engineering from the University of Washington, Seattle, in 1975. He did postdoctoral research at Providence Medical Center in Seattle, WA, for one year before being appointed a research bioengineer holding a joint appointment at the Institute of Applied Physiology and medicine. He became an assistant professor for the Bioengineering Program, The Pennsylvania State University, University Park in 1979 and was promoted to professor in 1989. He was a Distinguished Professor of Bioengineering at Penn State until September 1, 2002, when he joined the Department of Biomedical Engineering, University of Southern California, Los Angeles, as a professor. He has been the director of NIH Resource Center on Medical Ultrasonic Transducer Technology since 1997.

Dr. Shung is a fellow of the IEEE, the Acoustical Society of America, and the American Institute of Ultrasound in Medicine. He is a founding fellow of the American Institute of Medical and Biological Engineering. He has served for two terms as a member of the NIH Diagnostic Radiology Study Section. He received the IEEE Engineering in Medicine and Biology Society early career award in 1985 and coauthored a best paper published in the *IEEE Transactions on UFFC* in 2000. He is the distinguished lecturer for the IEEE UFFC Society for 2002–2003. He was elected an outstanding alumnus of Cheng-Kung University in 2001.

Fig. 1. Scanning electron microscope (a) cross-section and (b) top view of 12-μm PZT thick film.

Fig. 2.
 (a) Ferroelectric and (b) dielectric loss tangent (dashed line) and dielectric constant (solid line) of the 12- μm PZT thick film.

Fig. 3. Scanning electron microscope pictures (a) and (b) of the PZT film removed from the substrate with KOH.

Fig. 4.
Picture of the array pattern with 32 elements.

Fig. 5.
Fabrication flow of PZT film kerfless arrays.

Fig. 6.
Pictures of top electrodes of the thick-film linear arrays with an insulation layer.

Fig. 7.
Electric field effects and temperature effects in poling process.

Fig. 8. Modeled pulse-echo signals of (a) PZT film and (b) PZT-5H thin sheet kerfless array elements.

Fig. 9. Measured pulse-echo signals of (a) PZT film and (b) PZT-5H thin sheet kerfless array element.

TABLE I

Properties of PZT Thick Film and PZT-5H Ceramic.

Material	k_t	$\epsilon_{33}^S/\epsilon_0$	ρ (g/cm ³)	c (m/s)	$\tan \delta$ (%)
PZT film	0.34	600	6300	2900	3.4
PZT-5H	0.55	1470	7500	4560	2.0

k_t is the thickness mode electromechanical coupling coefficient, $\epsilon_{33}^S/\epsilon_0$ is the clamped dielectric constant, ρ is the density, c is the longitudinal wave velocity, and $\tan \delta$ (%) is the dielectric loss.

TABLE II

Modeling and Measured Results of Representative PZT Thick Film and PZT-5H Ceramic Elements.

Material	Center frequency (MHz)	Bandwidth (%) no matching	Bandwidth (%) with matching	Insertion loss (dB)
PZT film	120 (120)	45 (47)	60 (62)	41 (33)
PZT-5H	128 (123)	32 (30)	40 (44)	28 (21)

The values inside parentheses are the Piezo-CAD modeling results.