Published in final edited form as:

Appl Phys A Mater Sci Process. 2008 November 2; 2008: 161–163. doi:10.1109/ULTSYM.2008.0039.

PMN-PT single crystal thick films on silicon substrate for high-frequency micromachined ultrasonic transducers

J. Peng,

Department of Applied Physics and Materials Research Center, The Hong Kong Polytechnic University, Hong Kong, China, Fax: +852-233-37629

S.T. Lau,

Department of Applied Physics and Materials Research Center, The Hong Kong Polytechnic University, Hong Kong, China, Fax: +852-233-37629

C. Chao,

Department of Applied Physics and Materials Research Center, The Hong Kong Polytechnic University, Hong Kong, China, Fax: +852-233-37629

J.Y. Dai,

Department of Applied Physics and Materials Research Center, The Hong Kong Polytechnic University, Hong Kong, China, Fax: +852-233-37629

H.L.W. Chan,

Department of Applied Physics and Materials Research Center, The Hong Kong Polytechnic University, Hong Kong, China, Fax: +852-233-37629

H.S. Luo,

The State Key Laboratory of High Performance Ceramics and Superfine Microstructure, Shanghai Institute of Ceramics, Chinese Academy of Sciences, Shanghai 201800, China

B.P. Zhu,

Department of Biomedical Engineering and NIH Transducer Resource Center, University of Southern California, Los Angeles, CA 90089-1451, USA

Q.F. Zhou, and

Department of Biomedical Engineering and NIH Transducer Resource Center, University of Southern California, Los Angeles, CA 90089-1451, USA

K.K. Shung

Department of Biomedical Engineering and NIH Transducer Resource Center, University of Southern California, Los Angeles, CA 90089-1451, USA

J.Y. Dai: apdaijy@inet.polyu.edu.hk

Abstract

In this work, a novel high-frequency ultrasonic transducer structure is realized by using PMNPT-on-silicon technology and silicon micromachining. To prepare the single crystalline PMNPT-on-silicon wafers, a hybrid processing method involving wafer bonding, mechanical lapping and wet chemical thinning is successfully developed. In the transducer structure, the active element is fixed within the stainless steel needle housing. The measured center frequency and -6 dB bandwidth of the transducer are 35 MHz and 34%, respectively. Owing to the superior electromechanical coupling coefficient

(k_t) and high piezoelectric constant (d_{33}) of PMNPT film, the transducer shows a good energy conversion performance with a very low insertion loss down to 8.3 dB at the center frequency.

1 Introduction

High-frequency ultrasonic imaging has many clinical applications because of its improved image resolution (less than 10 μm) and is receiving acceptance as a clinical tool for the examination of skin [1,2], the arterial walls [3] and anterior chamber of the eye [4,5]. The development of high-frequency transducers has pushed the limits of ultrasonic imaging technology, giving diagnostic quality information about microscopic structures in living tissue. However, with operating frequency increasing up to 30 MHz, conventional transducer fabrication techniques are facing many difficulties in handling the miniaturized element and inter-element dimensions [6]. Piezoelectric micromachined ultrasound transducers (pMUTs) have thus been investigated as a promising new approach. By employing micromachining technologies, pMUTs offer advantages such as size reduction, batch production with high precision, repeatability and yield, low cost and possible realization of complete systems-on-a-chip [7].

Most of the reported pMUTs are exploiting the flexural vibration modes of micromachined membranes that work at relatively lower frequencies for airborne applications. To increase the resonant frequency, one has to reduce the dimension of the membrane [8]. Although a small membrane size below 50 μm has been achieved by using capacitive micromachined ultrasound transducer (cMUT) technology, it is still difficult for diaphragm-type micro transducers to achieve operation frequencies higher than 20 MHz. Besides, such a small active area will consequently lead to a lower efficiency of the transducer. Recently, Zhou et al. have reported a self-focused high-frequency pMUT-based on thickness mode vibration of a sputtered ZnO film [9]. High-frequency pMUTs using PZT [10] or PVDF [12] films as the functional layer to provide thickness mode vibrations have also been studied. The thickness mode resonance occurs when the thickness of the piezoelectric material is equal to odd multiples of one-half wavelength [11]. For the operating frequency over 20 MHz, one can reduce the thickness of transducer element, like PZT, down to 100 μm . As the relaxor-based ferroelectric single crystal $(1-x)\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3-x\text{PbTiO}_3$ (PMN-PT) possesses extraordinary piezoelectric properties [13,14], one would expect a much improved sensitivity of the transducer if the piezoelectric layers like ZnO or PVDF are replaced by single crystalline PMN-PT. Besides, due to its high relative permittivity, a 50 Ohm-impedance matching may be achieved with a reduced device size for PMN-PT-based transducer. This makes it possible to realize miniaturized transducers without degradation in performance, which is particularly interesting for intravascular applications.

In this work, we report the development of $\langle 001 \rangle$ -orientated PMN-30%PT thin film on-silicon wafer by using a hybrid method involving wet etching and micromachining. For high-frequency operation, thicknesses of PMN-PT layers are in the range from several microns to several tens of microns. With the PMNPT-on-Si wafer, a prototype pMUT with a center frequency of 35 MHz was fabricated and characterized.

2 Materials and methods

A 330 μm -thick PMN-PT single crystal with 30 mol% of PT, grown by a modified Bridgman method [14], was supplied by the Shanghai Institute of Ceramics. Chromium-gold (Cr/Au) electrodes with a thickness of 50/150 nm were coated on both sides of the sample surface by sputtering at 230°C. The sample was then poled under a DC field of 1 MV/m at 120°C for 15

minutes. To remove the trapped charges, the poled sample was short-circuited at 50°C for 24 hours.

To fabricate a pMUT working in the thickness mode, the electromechanical coefficient (k_t) and piezoelectric coefficient (d_{33}) are the important material parameters as they relate to the efficiency of the material in the energy conversion between electrical signal and mechanical vibration. Thus, for the material has high k_t and d_{33} value, it can be used to fabricate a transducer with good transmitting and receiving sensitivity. The electromechanical property of the poled PMN-PT was measured using an impedance analyzer (4294A, Agilent Technologies, Santa Clara, CA). Figure 1 shows the electrical impedance and phase of PMN-PT single crystal. Both resonant and anti-resonant peaks can be clearly observed at the frequency of 5.7 MHz and 7.1 MHz, respectively. The electromechanical coupling coefficient k_t can be determined by [15]

$$k_t = \sqrt{\frac{\pi}{2} \frac{f_r}{f_a} \tan\left(\frac{\pi}{2} \frac{f_a - f_r}{f_a}\right)} \quad (1)$$

where f_r is the resonant frequency and f_a is the anti-resonant frequency. The k_t is found to be 0.63. The piezoelectric coefficient d_{33} of the PMN-PT was measured using a ZJ-3B d_{33} meter (Institute of Acoustics, Academia Sinica, Shanghai, China). A brief list of the measured parameters for the PMN-PT single crystal plate is shown in Table 1.

Figure 2 shows the fabrication procedure of the PMN-PT pMUT. The poled PMN-PT single crystal was first bonded to a double-side polished silicon wafer ($\text{Si}_3\text{N}_4/\text{SiO}_2/\text{Si}/\text{SiO}_2/\text{Si}_3\text{N}_4$) by using a solvent-thinned epoxy-phenolic adhesive M-bond 610 (Vishay), which was cured at 50°C overnight. The PMN-PT layer was then lapped down to target thicknesses, ranged from several microns to several tens of microns, by using a hybrid method involving mechanical grinding and wet etching by HCl:HF solution in an ultrasonic bath [16]. For pMUT operating up to 35 MHz, the thickness of PMN-PT is close to 50 μm . To release the PMN-PT layer, the Si substrate with opening at the back was formed by KOH anisotropic wet etching, and the thin layers of M-bond 610/ Si_3N_4 were removed by O_2 plasma dry etching. The backside cavity was then filled with a conductive epoxy, E-solder 3022 (VonRoll Isola, New Haven, CT), as the bottom electrode as well as the backing material with acoustic impedance of about 6.23 MRayl. The silicon opening defined the actual aperture size of the pMUT (i.e. $0.7 \times 0.7 \text{ mm}^2$). The wafer was diced into $1.5 \times 1.5 \text{ mm}^2$ silicon dies. Each die carries a single PMN-PT element for subsequent device fabrication.

For making of prototype transducer, the silicon die was housed in stainless steel tubing with outer diameter of 2.5 mm. An electrical connector was fixed to the conductive backing using a conductive epoxy. A Cr/Au layer was sputtered across the PMN-PT layer and the stainless steel housing to form the ground plane connection. Finally, a layer of vapor deposited parylene with a thickness of 10 μm was coated on the transducer surface as an acoustic matching layer, which is used to improve ultrasonic energy transmission between the piezoelectric material and the load medium. The acoustic impedance of the matching layer is 2.59 MRayl.

Figure 3 is the photograph of finished PMN-PT pMUT. A 50 Ω coaxial cable with the shortest practical length (~20 cm) was used to connect the pMUT to the pulser/receiver in the pulse-echo measurement. To optimize the acoustic design of the transducer including the front-face matching and backing, the KLM model-based simulation software PiezoCAD (Sonic Concepts, Woodinville, WA) was used. The piezoelectric properties of PMN-PT may be degraded due to mechanical polishing and heat treatment during the device fabrication. To

enhance the transducer performance, the PMN-PT element was repoled under DC 180 V at room temperature for 15 minutes.

To evaluate the resonance characteristics of the transducer, the electrical impedance of the transducer was measured as a function of frequency by 4294A impedance analyzer. The performance of the transducer was tested using a pulse-echo response arrangement [17]. The setup consisted of measuring the received echo from the reflection of a flat quartz target in a deionized water bath. A Panametrics pulser/receiver (5900PR, Olympus NDT, Waltham, MA) was used to excite the transducer and receive the echo waveform. The received echo signal was recorded by the digital oscilloscope with 50 Ω coupling. The FFT math feature on the oscilloscope was used to display the Fourier transform of the received waveform. From the measured frequency spectra of the transducers, the following parameters of the transducer can be calculated by

- a. Lower and upper -6 dB frequencies (f_l and f_u): the frequencies at which the magnitude of the amplitude in the spectrum is 50% (-6 dB) of the maximum.
- b. Center frequency:

$$f_c = \frac{f_l + f_u}{2}. \quad (2)$$

- c. -6 dB bandwidth:

$$BW = \frac{f_u - f_l}{f_c} \times 100\%. \quad (3)$$

For measuring the two-way insertion loss (or the relative pulse-echo sensitivity), the transducer was excited by a multi-cycle sine wave burst from a function generator AFG 2020 Sony/Tektronix, Beaverton, OR) with 50 Ω coupling. The peak amplitude of the received echo from the transducer was measured by an oscilloscope with 1 M Ω coupling. After disconnecting the transducer, the peak amplitude of the burst waveform from the function generator was measured by an oscilloscope with 50 Ω coupling. The receive pressure/voltage lost due to absorption in the quartz target was compensated by [18]

$$P_I = P_R \left(\frac{Z_Q + Z_w}{Z_Q - Z_w} \right) \quad (4)$$

where P_I is the incident pressure of the pulse, P_R is the reflected pressure, and Z_Q and Z_w are the acoustic impedance of the quartz (15.3 MRayl) and water (1.5 MRayl), respectively. Since voltage is analogous to pressure, the incident voltage can be directly calculated using (4). The signal loss due to attenuation in the water bath was also compensated for using an attenuation of $2.2 \times 10^{-4} f^2$ dB/mm (with f in MHz) [18].

3 Transducer characterization

The impedance and phase spectrum of the PMN-PT pMUT were measured in air using an impedance analyzer and the results are shown in Fig. 4. The resonant frequency f_r and the anti-resonant frequency f_a were approximately at 28.5 MHz and 35.8 MHz, respectively, and the electrical impedance was about 62 Ω at resonance. The effective electromechanical coupling coefficient k_t' was calculated to be 0.64, which was close to the k_t of PMN-PT bulk single

crystal plate (0.63), implying a very high energy conversion efficiency of the transducer. The relative permittivity at 1 kHz was found to be 2213 at room temperature.

The pulse-echo response of the PMN-PT pMUT was characterized in a water tank at room temperature. Figure 5 shows the pulse-echo response and frequency spectrum of the pMUT. According to (2) and (3), the center frequency of the pMUT is found to be 35 MHz and the -6 dB bandwidth is 34%. A ring-down is observed in the echo waveform, leading to relatively low bandwidth. To improve the transducer bandwidth, multiple matching layers can enhance ultrasonic energy transmission across the element-load medium boundary better than does a single layer. However, this may increase the production cost. Thus, two layers are recommended by adding a second matching layer before the parylene on the front side of the transducer. Figure 6 shows the insertion losses IL measured at different frequencies. The minimum IL is found to be 8.3 dB at the center frequency. Using the pulser/receiver with 1 μ J energy setup, the maximum peak-to-peak output voltage of the unamplified echo signal was 1.1 V (see Fig. 5). These results show that the PMN-PT pMUT has a high sensitivity due to its excellent electromechanical properties.

4 Conclusion

This paper describes the development and fabrication of a high-frequency (35 MHz) ultrasonic transducer with an aperture size of 0.7×0.7 mm² by micromachining of single crystalline PMN-PT on silicon substrate. The PMN-PT thin layer exhibits excellent electromechanical properties which are comparable to those of bulk PMN-PT single crystals. The transducer has a -6 dB bandwidth of 34% and a minimum two-way insertion loss of 8.3 dB at the center frequency. This insertion loss is significantly better than what can be obtained using devices with the conventional piezoelectric materials (such as PZT and PVDF). A further improvement in both bandwidth and insertion could be achieved through the use of a double-matching layers scheme.

Acknowledgments

This work was supported by the Hong Kong Innovative Technology Council (Project No. K-ZP21), the Centre for Smart Materials of the Hong Kong Polytechnic University, NIH Grant No. P41-EB2182.

References

1. Passmann, C.; Emert, H. Proc. IEEE Ultrason. Symp; 1994. p. 1661
2. Turnbull DH, Starkoski BG, Harasiewicz KA, Semple IL, From L, Gupta AK, Sauder DN, Foster FS. J Ultrason Med 1995;21:71.
3. Bruce C, Packer D, Belohlavek M. J Am Soc Echocardiogr 2000;13:788. [PubMed: 10936825]
4. Pavlin, J.; Stuart, FS. Ultrasound Biomicroscopy of the Eye. Springer; Berlin: 1995.
5. Ritter TA, Shung KK, Cao WW. IEEE Trans Ultrason Ferroelectr Freq Control 2002;49:217. [PubMed: 11885679]
6. Cannata JM, Ritter TA, Chen WH. IEEE Trans Ultrason Ferroelectr Freq Control 2003;50:1548. [PubMed: 14682638]
7. Fleischman A, Modi R, Nair A. Sens Actuators A Phys 2003;103:76.
8. Gaucher, P. Piezoelectric micro-electro-mechanical systems for acoustic applications. In: Setter, N., editor. Piezoelectric Materials in Devices. Vol. Chap. 13. Ceramics Laboratory, EPFL Swiss Federal Institute of Technology; 2002.
9. Zhou QF, Sharp C, Cannata JM, Feng GH, Kim ES, Shung KK. Appl Phys Lett 2007;90:113502.
10. Zhang QQ, Djuth FT, Zhou QF. Ultrasonics 2006;44:711.
11. Gottlieb EJ, Cannata JM, Hu CH, Shung KK. IEEE Trans Ultrason Ferroelectr Freq Control 2006;53:1037. [PubMed: 16764457]

12. Shung, KK. Diagnostic Ultrasound-Imaging and Blood Flow Measurements. Vol. Chap. 3. CRC Press; Boca Raton: 2006. p. 47
13. Service RE. Sci 1997;275:1878.
14. Luo H, Xu G, Wang P, Yin Z. Ferroelectrics 1999;231:97.
15. Standard. 1987. IEEE Standard on Piezoelectricity; p. 176-1987.
16. Peng J, Chao C, Dai JY, Chan HLW, Luo H. Mater Lett 2008;62:3127.
17. Selfridge, AR. PhD Dissertation. Stanford University; Stanford, CA: Jul. 1983 The design and fabrication of ultrasonic transducers and transducer arrays.
18. Lockwood GR, Turnbull DH, Foster FS. IEEE Trans Ultrason Ferroelectr Freq Control 1994;41:231–235.

Fig. 1.
Electrical impedance (*solid line*) and phase (*dot line*) of PMN-PT single crystal plate

Step 1: Bonding PMN-PT on the Si-wafer by M-bond 610, and lapping the PMN-PT down to $50 \mu\text{m}$ by a hybrid method.

Step 2: Opening the backside window by micromachining. The thick Si layer is removed by KOH and the layers of M-bond 610/ Si_3N_4 is removed by O_2 plasma dry etching.

Step 3: Filling the backside cavity with conductive E-solder 3022 and then dice the wafer into $1.5 \times 1.5 \text{ mm}^2$ silicon dies.

Fig. 2.
Fabrication process of the PMN-PT pMUT

Fig. 3.
Photograph of the prototype PMN-PT pMUT

Fig. 4.
Measured electrical impedance magnitude and phase angle for the PMN-PT pMUT

Fig. 5.

The measured pulse-echo response and frequency spectrum of PMN-PT pMUT

Fig. 6.
The insertion losses of PMN-PT pMUT measured at different frequencies

Table 1Material properties of $\langle 001 \rangle$ -orientated PMN-30%PT single crystal

Electromechanical coupling coefficient k_t	0.63
Piezoelectric charge coefficient $d_{33}(\times 10^{-12} \text{ C/N})$	1100
Relative permittivity at 1 kHz ϵ_{33}^T	1380
Dissipation factor $\tan \delta$ at 1 kHz	0.008
Density (kg/m^3)	8100
Longitudinal wave velocity (m/s)	3600
Acoustic impedance (MRayl)	29.2