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High Frequency PMN-PT 1–3 Composite Transducer for Ultrasonic Imaging Application

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Abstract

Development of PMN-PT single crystal/epoxy 1–3 composites for high-frequency ultrasonic transducers application is presented. The composite was fabricated by using a DRIE dry etching process with a 45% volume fraction of PMN-PT. A 35 MHz ultrasound flat transducer was fabricated with the composite, which was found to have an effective electromechanical coupling coefficient of 0.81, an insertion loss of 18 db, and a –6 dB bandwidth as high as 100%. Tungsten wire phantom image shows that the transducer had an axial resolution of 30 μm , which was in good agreement with the theoretical expectation. The initial results showed that the PMN-PT/epoxy 1–3 composite has many attractive properties over conventional piezoelectric materials for medical imaging applications.

Keywords

PMN-PT; 1–3 composite; Ultrasonic transducer

I. Introduction

Piezoelectric 1–3 composite ultrasonic transducers have been widely used in medical ultrasonic imaging due to their superior performance in comparison with conventional monolithic ceramics and polymers [1, 2]. Piezoelectric 1–3 composites provide many advantages including higher electromechanical coupling coefficient (k_t), lower electric and mechanical loss and lower acoustic impedance. By proper selection of the monolithic ceramics, polymer, and volume fractions, the composite properties can be tailored to meet specific device requirements.

Moreover, because of the large bandwidth and high sensitivity, PMN-PT transducers can be used for high-frequency (>30 MHz) ultrasonic imaging and Doppler applications [3]. Hence, there is a need to develop PMN-PT 1–3 composite transducers that can operate efficiently at high frequencies. In order to fabricate such transducers, the control and reduction of spatial scale of the ceramic pillars inside the composite is required. The traditional technique for 1–3 composite fabrication such as dice-and-fill has limited utility in reducing the diameter of pillars due to the brittleness of the ceramics. Recently, the TRS group and Boston Scientific

demonstrated that it was possible to fabricate composite transducers with a PC-MUT fabrication process. It provided a basic guide to design 1–3 composite transducers [4–6].

In this work, PMN-PT 1–3 composite was fabricated with a novel etching-and-filling technique: a DRIE dry etching process with a profile angle of 85° was developed for patterning PMN-PT single crystal; subsequently the kerfs were filled with Epotek-301 epoxy forming a 1–3 structure. A high frequency (35 MHz) single element flat transducer with 45% volume fraction of composite was fabricated. The impedance and the pulse-echo, characteristics of this 1–3 composite transducer were measured and compared with theoretical predictions. Finally, the image of a 20 μm diameter tungsten wire phantom was acquired, and the resolution of the composite transducer was estimated.

II. Design and Methods

PMN-PT single crystals were supplied by HC Materials Corps (Urbana, IL). The properties of the samples are shown in Table I. The 1–3 composite was fabricated using a novel etch-and-fill technique according to the PC-MUT fabrication process [6]. The fabrication process is shown in Fig. 1. PMN-PT wafers were lapped on both sides and polished on one side. A photo-lithography-based method was applied to transfer design patterns onto the polished surface. A layer of Cr/Au (500 Å/1000 Å) was first patterned onto the 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 7 μm thick Ni hard mask through the openings of the photoresist pattern. After electroplating, the photoresist was stripped off using Acetone. The ICP (Inductively Coupled Plasma)-RIE (Reactive Ion Etching) dry etching technique was used to etch the single crystal. The area underneath the nickel hard mask was protected; the rest of the area was etched off by chlorine gas plasma. Fig. 2 shows the SEM photos of an etched crystal. The measurements show that the thickness of the elements is $\sim 40 \mu\text{m}$, the sidewall angle of the elements is $> 85^\circ$. The kerfs were infiltrated with Epo-Tek 301–2 epoxy (Epoxy Technology, Billerica, MA). Before filling the kerfs, a MARCH plasma cleaning system was used to make the array surface hydrophilic, so that it is easier to be fill micro-scale kerfs with epoxy. A degassing process was conducted right after filling. The epoxy was cured at the room temperature over night followed by 2 hours curing at an elevated temperature. After curing, the extra epoxy and residual nickel were carefully lapped away with fine sandpapers followed by 3- μm Al_2O_3 powder. Finally a layer of Cr/Au (500 Å/1500 Å) was patterned as the electrode.

A physical model of the thickness-mode oscillation in piezoelectric composite plates [7] shows that the electromechanical coupling k_t of the composite is larger than pure ceramics and reaches an almost constant value at the volume fraction of 40%–80%. The acoustic impedance of the composite increases with the increase of volume fraction. Thus, a volume fraction of 40%–60% is adopted, making the acoustic impedance of the composite (15–20 MRayls at the volume fraction of 40%–60%) much lower than that of pure ceramic (> 30 MRayls). Here the width of the PMN-PT rod is designed to be 10 μm ; the kerf between PMN-PT rods is designed to be 5 μm . With such a pattern, the volume fraction of the PMN-PT is calculated to be 45%; thus the acoustic impedance of the composite is 18 MRayls.

1–3 composite PMN-PT single element flat transducers with center frequencies of 35 MHz are designed with the simulation software PiezoCAD (Sonic Concepts, Woodinville, WA). The size of the piezo-material is designed to $0.9 \times 0.9 \text{ mm}^2$, which matches the standard 50 Ω load of the pulser/receiver electronics. Because of the lower acoustic impedance of the composite material (18 MRayl), only one parylene layer (Specialty Coating Systems,

Indianapolis, IN) is selected as the matching layer (2.5 MRayls). In addition, to further improve the bandwidth of the transducers, conductive epoxy Esolder-3022 (VonRoll Isola, New Haven, CT) (5.9 MRayls) is used as backing material. The simulation results are shown in Fig. 3.

The transducers were fabricated following the procedure reported by Cannata et al [8]. The composite plate was first lapped down to the design thickness and cured by a conductive back layer, then was housed using insulating epoxy (Epotek 301, Epoxy Technology Inc., Billerica, MA) within brass tubes. An additional chrome/gold electrode was sputtered across the composite layer and the housing to form the ground connection. Finally, parylene was evaporated onto the front surface as the second matching layer. A SMA connector was then mounted. After fabrication, the transducer was poled in air at 70°C temperature with an electric field of 2 V/μm for about 5 minutes. The structure of the transducer is shown in Fig. 4.

III. Results and Discussion

With a 1715 LCR Meter (QuadTech Inc, Sussex, WI), the loss and the capacitance of the transducer were measured. The dielectric loss was found to be 0.040 and the capacitance 337 pf. According to the equation:

$$\epsilon_r = \frac{C \times d}{A \times \epsilon_0} \quad (1)$$

where C is the capacitance, d is the thickness of the element, A is the size of the element, ϵ_0 is found to be 8.85×10^{-12} f/m. The calculated dielectric constant is 1503, which is close to the theoretical value (1600).

The impedance and phase diagrams of the 1–3 composite were measured in air with a 4294A precision impedance analyzer (Agilent Technologies Inc. Philadelphia, PA). As shown in Fig. 5, the transducer has a clean thickness resonance of around 40 MHz and electrical impedance of around 25 ohms at the resonance frequency. The electromechanical coupling coefficient k_t can be determined from the following equation according to the IEEE standard [9]

$$K_t = \sqrt{\frac{\pi}{2} \times \frac{f_s}{f_p} \times \tan\left(\frac{\pi}{2} \times \frac{f_p - f_s}{f_p}\right)} \quad (2)$$

where f_s is the resonant frequency at which the conductance reaches the minimum and f_p is the parallel resonant frequency at which the resistance reaches the maximum. In Fig. 5, f_s is 32 MHz, and f_p is 51 MHz. According to equation (2), k_t was calculated to be 0.81. It is substantially higher than that of PMN-PT transducer ($k_t = 0.58$) [3].

A pulse-echo method [10] was employed to characterize the transducer performance with a Panametrics model 5910PR pulser-receiver (Olympus NDT, Waltham, MA) and a LeCroy LC534 oscilloscope (LeCroy Corp., Chestnut Ridge, NY). Distilled water at room temperature was used as the propagation medium. The pulse-echo response (Fig. 6) of the transducer was obtained by placing a flat quartz target at a distance of 1.5mm in front of the transducer. The response echo exhibited an amplitude of about 1.1 V. The frequency spectrum (Fig. 6) exhibited a center frequency of 32 MHz for this composite transducer. The center frequency of the transducer was a little lower than that predicted by model. This

discrepancy was most likely caused by either thickness variability in the parylene layers, or frequency attenuation in water. From the frequency spectrum, the fractional bandwidth (BW), which was the key property of the transducer, was estimated to be 100%.

Insertion loss was measured according to the method reported by Snook et al. [11]. A multi-cycle sinusoidal pulse was produced using a Sony/Tetronix (Beaverton, OR) model AFG2020 arbitrary function generator. The amplitude of the input sinusoid signal was set to 5 V at the transducer's center frequency, and recorded with a 50 Ω coupling on the Lecroy oscilloscope. The transducer was then connected to the function generator with the oscilloscope, and the coupling was changed to 1 M Ω . The echo signal amplitude (signal averaging) was measured at 2 μ s according to a sound speed of 1540 m/s. This distance was beyond the natural focal point of the transducer due to ringing noise from the function generator. The signal loss from the attenuation in water is $(2.2 \times 10^{-4} \text{ dB/mm}\cdot\text{MHz}^2)$, and the transmission into the quartz target was compensated in the final insertion loss calculation. Insertion loss was measured to be around 18 dB.

A system (Fig. 7) was set up to measure the beam profile. In this study, the transducer was driven by a motor controller DMC-1802 (Galil Motion Control Inc., Mountain View, CA), which also generated a signal to trigger data acquisition. The Panametrics 5900PR pulser/receiver was used to excite the transducer. CompuScope-12400 data acquisition card (Gage Applied Technologies, Lockport, IL) at a sampling frequency of 400 MHz and transferred to the PC. A tungsten wire phantom was used as the target. The wire phantom contained 5 wires separated laterally by 0.65 mm and axially by 1.55 mm. Each of the wires was 20 μ m in diameter. Both the transducer and the wire phantom were placed in a water tank filled with distilled water. The pulse-echo image was acquired as the transducer scanned across the wire phantom placed in degassed water. The gray scale image (Fig. 8) was displayed by software Matlab (Mathworks inc, Natick, MA), with a gain of 40 dB. The theoretical value of axial resolution at -6 dB can be calculated according to equation (3):

$$R_{ax} = \frac{C}{2 \times BW} \quad (3)$$

where C is the velocity at the loading medium, BW is the bandwidth of the transducer. Thus the theoretical value of axial resolution is 24 μ m. The image results show an axial resolution of 30 μ m at -6 dB, which is in good agreement with the theoretical expectation.

IV. Conclusion

PMN-PT/epoxy 1-3 composites with volume fraction 0.45 was designed and fabricated with a DRIE dry etching processes. Ultrasonic transducers were subsequently fabricated using the PMN-PT /epoxy 1-3 composite. A transducer with a center frequency of 35 MHz developed from a $V_f = 0.45$ composite had a coupling coefficient K_t of 0.81, and a bandwidth of 100%. The measured results show that the transducer has an axial resolution of 30 μ m. PMN-PT 1-3 composite high-frequency single-element focused transducers and linear arrays are currently under development in an effort to further improve the spatial resolution of ultrasonic images. Results showed that PMN-PT/epoxy 1-3 composite is a promising candidate for high frequency medical ultrasonic transducer applications.

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Figure 1.
Fabrication process of the PMN-PT 1–3 composite.

Figure 2.
SEM photos of the etched crystal.

Figure 3.
Simulation of the PMN-PT 1–3 composite transducer.

Figure 4.
Structure of a PMN-PT/epoxy 1–3 composite transducer.

Figure 5.
The impedance and phase diagrams of the PMN-PT/epoxy 1–3 composite transducer.

Figure 6. Pulse-echo response and the Frequency spectrum of PMN-PT/epoxy 1–3 composite transducer.

Figure 7.
Block diagram of beam profile measurement system.

Figure 8. Wire-phantom images of the PMN-PT 1–3 composite transducer with dynamic range of 40 dB.

Table 1

Properties of PMN-PT single crystals

Property	PMN-PT
Electromechanical coupling coefficient (k_t)	0.58
Piezoelectric constant d_{33} of wafer (pC/N)	1301
Relative clamped dielectric constant (ϵ^S/ϵ_0)	797
Dielectric loss	0.0036
Density (g/cm ³)	8.00
Longitudinal wave velocity (m/s)	4608
Acoustic impedance (MRayl)	32.6
Coercivity (kV/cm)	2.5