Single Crystal PMN-0.33PT/Epoxy 1-3 Composites for Ultrasonic Transducer Applications

Kei C. Cheng, Helen L. W. Chan, Chung L. Choy, Qingrui Yin, Hasou Luo, and Zhiwen Yin

***Abstract*—Lead magnesium niobate-lead titanate 0.67Pb $(Mg_{1/3}Nb_{2/3})O_3$-0.33PbTiO$_3$ (PMN-0.33PT, abbreviated as PMN-PT) single crystals were used to fabricate PMN-PT/epoxy 1-3 composites with different volume fractions of PMN-PT ranging from 0.4 to 0.8. The electromechanical properties of the 1-3 composites were determined by the resonance technique. Theoretical modeling of the 1-3 composites matched quite well with the measured material properties. It was demonstrated that the thickness electromechanical coupling coefficients of the composites could reach as high as 0.8. A 2.4 MHz plane ultrasonic transducer was fabricated using a PMN-PT/epoxy 1-3 composite with 0.37 volume fraction of PMN-PT. It shows a −6 dB bandwidth of ∼61% and an insertion loss of −14 dB.**

I. Introduction

The development of relaxor-based single crystals such as lead magnesium niobate-lead titanate (PMN-PT) and lead zinc niobate-lead titanate (PZN-PT) has promising potential for ultrasonic transducer and actuator applications [1]–[8]. The single crystals exhibit high piezoelectric coefficient ($d_{33} > 2000$ pC/N) and electromechanical coupling factor ($k_{33} \sim 0.9$); commonly used piezoceramic PZT provides d_{33} of ∼600 pC/N and k_{33} of ∼0.75. The single crystals with these superior properties can provide better sensitivity and bandwidth for medical imaging applications [2], [4], [5]. However, for ultrasonic imaging and hydrophone applications, the relatively high acoustic impedance of the single crystals (approximately 30 Mrayls) raises a problem of acoustic impedance mismatch with that of human tissue (1.5 Mrayls) and water. In order to alleviate this problem, the single crystal is incorporated into a polymer matrix to form 1-3 composite. The 1-3 composites shown in Fig. 1 consists of PMN-PT rods embedded in an epoxy matrix. As the relatively softer polymer matrix cannot clamp the vibration of the PMN-PT rods, the PMN-PT rods inside the composites are relatively free to vibrate. Hence, the thickness electromechanical coupling coefficient k_t of the composites can approach the k_{33} of the PMN-PT [9]–[13]. The k_{33} is about 0.8–0.9 for PMN-PT rod, and k_t is about 0.6 for PMN-PT disc. In other words, using 1-3 PMN-PT composites can improve the energy conversion efficiency of the transducer. Furthermore, the electromechanical properties of the 1-3 composites also can be tailored by varying the volume fraction and periodicity to optimize the transducer performance.

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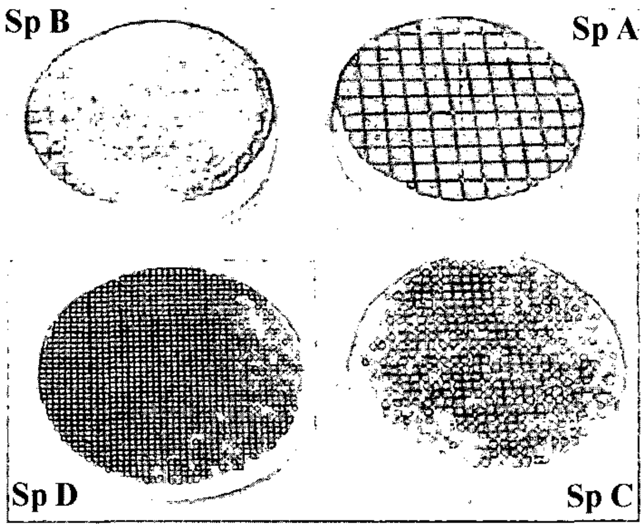

Fig. 1. Photograph of PMN-PT/epoxy 1-3 composites.

II. Material Fabrication

The relaxor ferroelectric PMN-PT Z-cut single crystal discs of 10-mm diameter and 3-mm thickness were grown in the Shanghai Institute of Ceramics, Chinese Academy of Sciences (SICCAS), Shanghai, China, by a modified Bridgeman method [6]. As stated in [8], the single crystals poled along the ⟨001⟩ direction possess the maximum piezoelectric response. It is reported that [8] poled single crystals have a pseudo-tetragonal 4-mm macroscopic symmetry even though its microscopic symmetry is 3 m [8]. The calculated material properties presented in this paper are based on the 4-mm symmetry. Before poling, Cr-Au electrodes were applied to the flat surfaces of the PMN-PT discs by magnetron sputtering. They were poled in oil along their thickness direction by applying a direct current (DC) field of 10 kV/cm at 110°C for 10 minutes, and the

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TABLE I
PROPERTIES OF PMN-PT/EPOXY 1-3 COMPOSITES.

	Sp A	Sp B	Sp C	Sp D
Thickness h (mm)	2.60	2.72	1.71	0.75
Lateral rod size L (mm)	0.832	0.488	0.33	0.16
Width of epoxy d (mm)	0.108	0.105	0.090	0.087
No. of cuts	10	16	25	40
Volume fraction	0.77	0.62	0.48	0.37
Piezoelectric d_{33} (pC/N) of the PMN-PT single crystal before cutting	1980	1840	1900	2000
Density (kg/m^3)	6652	5411	4419	4460

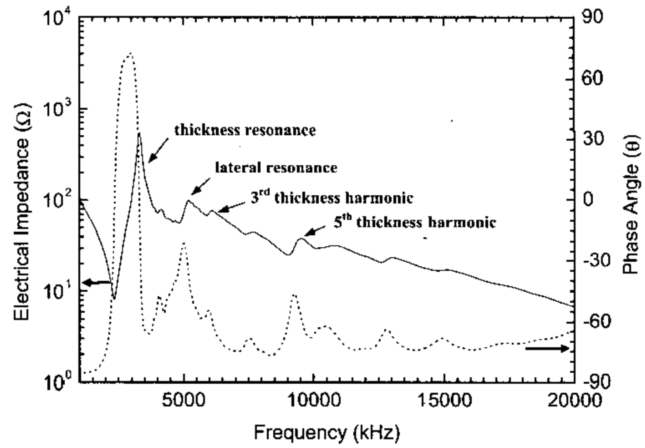

Fig. 2. Electrical impedance Z and phase angle θ against frequency spectrum (1 MHz–20 MHz) for a PMN-PT/epoxy 1-3 composite disk with a 0.37 volume fraction of PMN-PT.

electric field was kept on until the samples were cooled to room temperature.

Four samples of PMN-PT/epoxy 1-3 composites with volume fractions of 0.4 to 0.8 as shown in Fig. 1 were made by a dice-and-fill technique [11]–[13]. Poled single crystals were cut in one direction using an automatic dicing saw (DAD 321, Disco Corporation, Tokyo, Japan) with an 80 μm thick blade. Epoxy LW5157 with a hardener HY5159 (in 10:1 ratio) supplied by Ciba-Giegy Ltd., USA, was used to fill the grooves in the single crystals. After the epoxy had cured, a second set of cuts perpendicular to the first direction was made. If reticulated cuts were made in both directions before casting the epoxy, the PMN-PT rods may break because they are very fragile. It was found that, after filling the first set of grooves, if a thin layer of epoxy was left on top of the crystal, the chance of chipping the crystal during the second set of cuttings could be greatly reduced. After filling the second set of cuts by epoxy, the composite was allowed to cure at 40°C in an oven for 12 hours in order to develop optimum properties. Excess epoxy was ground away with wet and dry papers (no. 800 and 1000, Kovax, Tokyo, Japan). Repoling the composites was implemented in case they were depoled during the cutting.

III. PROPERTIES OF PMN-PT/EPOXY 1-3 COMPOSITES

The composites fabricated in this study all have fine polymer width so that they can be considered as homogeneous piezoelectric materials. Piezoelectric d_{33} value and the density of the composites and the single crystals were measured and are shown in Table I. The PMN-PT single crystals used to fabricate the PMN-PT/epoxy 1-3 composites have high piezoelectric coefficient d_{33} of ~2000 pC/N as measured by a Model ZJ-3D Piezo-d_{33} meter supplied by the Beijing Institute of Acoustics Academia Sinica, China.

The electromechanical properties of PMN-PT/epoxy 1-3 composites were determined at room temperature following the *IEEE Standards on Piezoelectricity* [14]. Thickness electromechanical coupling coefficient k_t, mechanical quality factor Q_M, and elastic stiffness c_{33}^D were determined by measuring the resonant and antiresonant frequencies of the thickness mode resonance in the composite disc. From the electrical impedance vs frequency plot (Fig. 2), a strong thickness resonance with its third and fifth harmonics can be observed. These resonant frequencies were measured with an HP 4294A (Agilent, Palo Alto, CA) impedance analyzer. In order to avoid mode coupling, which adversely affects the measurement, the thickness to width or aspect ratio of the PMN-PT rods inside the composite samples was higher than 3. A weak vibration mode f_L arisen from the lateral vibration of the PMN-PT rods [10], [13] was observed. Due to blade vibration, the resultant polymer width in the composites varied from 85 μm to 100 μm, which was larger than the thickness of the blade. However, the estimated stopband resonances will be >10 MHz [10], [13] that are not observed in the frequency range of interest. Other parameters such as the acoustic velocity V_3^D and the relative permittivity $\varepsilon_{33}/\varepsilon_0$ at 1 kHz were also determined.

The measured piezoelectric, dielectric, and electromechanical properties of the epoxy and the PMN-PT single crystals are given in Tables II and III [15], respectively. The theoretical properties of the 1-3 composites as a function of volume fraction are calculated by the modified series and parallel model [11], [12]. The electromechanical coupling coefficient k_t in the composite can be determined by (1):

$$k_t^2 = \frac{\pi}{2}\frac{f_r}{f_a}\tan\left(\frac{\pi}{2}\frac{f_a - f_r}{f_a}\right), \qquad (1)$$

where f_r is the resonant frequency and f_a is the antiresonant frequency and assumed that f_r ~ frequency of minimum impedance and f_a ~ frequency of maximum impedance, as the PMN-PT single crystals and the composites have low dielectric loss [14]. Experimentally, it was found that the dielectric loss factor $\tan\delta$ is less than 1% for all the composites.

As discussed before, k_t in 1-3 composites can be enhanced by using PMN-PT rods embedded in the nonpiezo-

TABLE II
ROOM-TEMPERATURE MATERIAL PROPERTIES OF EPOXY.

Density	ρ	(kg/m^3)	1200
Relative permittivity	$\varepsilon/\varepsilon_0$		4.21
Dielectric loss factor	$\tan\delta$		0.005
Longitudinal wave velocity	v_L	(m/s)	2658
Shear wave velocity	v_S	(m/s)	1416
Longitudinal acoustic impedance	Z_L	(Mrayl)	3.19
Shear acoustic impedance	Z_S	(Mrayl)	1.70
Longitudinal stiffness	c_{11}	(10^{10}N/m^2)	0.85
Shear stiffness	c_{44}	(10^{10}N/m^2)	0.24
Stiffness in x-y plane	c_{12}	(10^{10}N/m^2)	0.37
Longitudinal compliance	s_{11}	(10^{-12}m^2/N)	159.61
Shear compliance	s_{44}	(10^{-12}m^2/N)	415.57
Compliance in x-y plane	s_{12}	(10^{-12}m^2/N)	−48.19
Shear modulus	G	(10^{10}N/m^2)	0.24
Bulk modulus	K	(10^{10}N/m^2)	0.53
Young's modulus	Y	(10^{10}N/m^2)	0.63
Mechanical quality factor	Q_M		24.5
Poisson's ratio	σ		0.30

TABLE III
ROOM-TEMPERATURE MATERIAL PARAMETERS OF THE PMN-PT SINGLE CRYSTALS.

Density	ρ	(kg/m^3)	7900
Relative permittivity	$\varepsilon/\varepsilon_0$		5214
Dielectric loss factor	$\tan\delta$		0.003
Piezoelectric coefficient	d_{33}	(pC/N)	1980
Electromechanical coupling coefficient	k_t		0.6
Elastic stiffness	c_{33}^D	(10^{10}N/m^2)	17.2
Elastic stiffness	c_{33}^E	(10^{10}N/m^2)	11.0
Mechanical quality factor	Q_M		41

electric passive epoxy matrix. The electromechanical coupling coefficient k_t of the composite as shown in Fig. 3 can reach as high as 0.8, which is higher than that of a PMN-PT disc ($k_t \sim 0.61$), but it is still lower than the value of a single PMN-PT rod ($k_{33} \sim 0.9$). The reason may be because the epoxy exerts a certain degree of lateral clamping on the PMN-PT rods and modify its behavior as a totally free rod. In general, using a softer polymer matrix can reduce the clamping to the single crystal and increase the electromechanical coupling coefficient. However, the use of highly compliant polymers will lower the stopband frequency and provide little support during fabrication and breakage of the rods will occur [4].

Plot of c_{33}^D in Fig. 4 shows that c_{33}^D linearly increases with the volume fraction. The elastic stiffness c_{33}^D can be calculated by (2):

$$c_{33}^D = \rho\,(2tf_a)^2, \tag{2}$$

where ρ is a density and t is the thickness of the composite.

Fig. 5 shows that the relative permittivity $\varepsilon_{33}/\varepsilon_0$ measured at 1 kHz also linearly increases with the volume fraction ϕ (for $\phi > 0.3$). In Fig. 6, it is seen that Q_M of the composites of all the volume fractions was about 10 to 20,

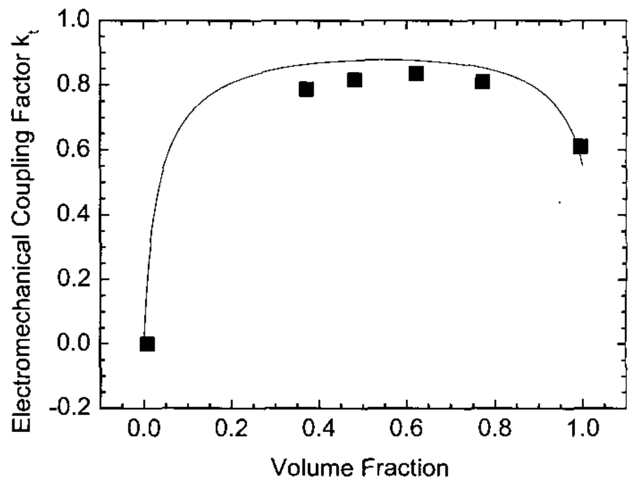

Fig. 3. Electromechanical coupling coefficient k_t as a function of single crystal volume fraction.

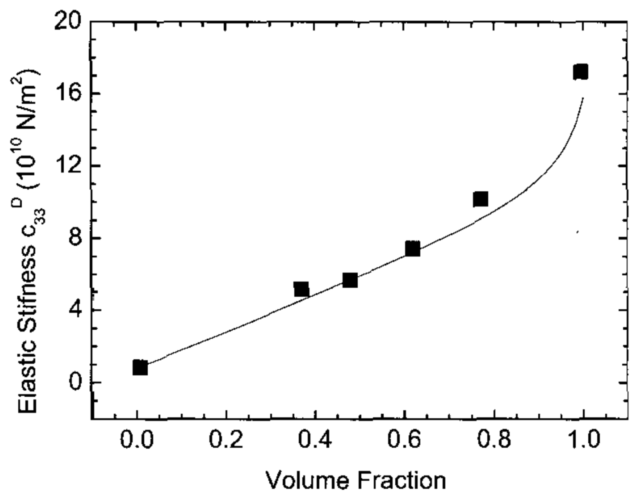

Fig. 4. Elastic stiffness c_{33}^D as a function of single crystal volume fraction.

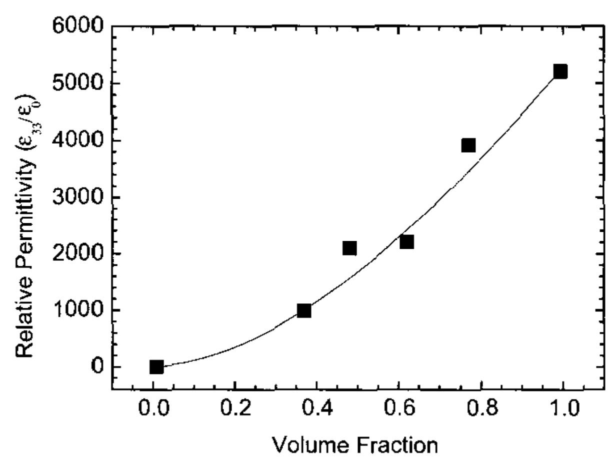

Fig. 5. Relative permittivity $\varepsilon_{33}/\varepsilon_0$ as a function of single crystal volume fraction.

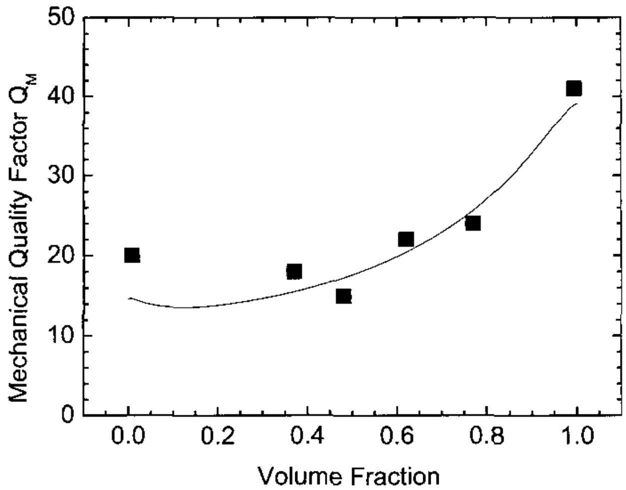

Fig. 6. Mechanical quality factor Q_M as a function of single crystal volume fraction.

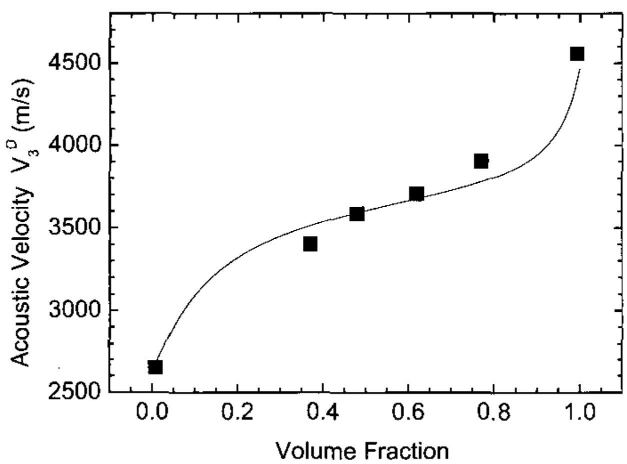

Fig. 7. Acoustic velocity V_3^D as a function of single crystal volume fraction.

which is quite close to that of the polymer. The mechanical quality factor Q_M was calculated by (14):

$$Q_M = \frac{1}{2\pi f_{rr} Z_m (C_0 + C_1)(f_{aa}^2 - f_{rr}^2)}, \quad (3)$$

where C_1 is the motional capacitance and C_0 is the clamped electrical capacitance, respectively. Z_m is the minimum impedance, f_{rr} and f_{aa} are the resonant and antiresonant frequencies at the radial mode resonance of the PMN-PT disc, respectively.

The acoustic velocity V_3^D also increases almost linearly with the volume fraction ϕ (for $0.3 < \phi < 0.9$) as shown in Fig. 7.

From Figs. 3–7, it is seen that the modified series and parallel model [11], [12], although simple, can give quite accurate prediction to the materials properties of 1-3 composites with $\phi > 0.2$ in the absence of mode coupling.

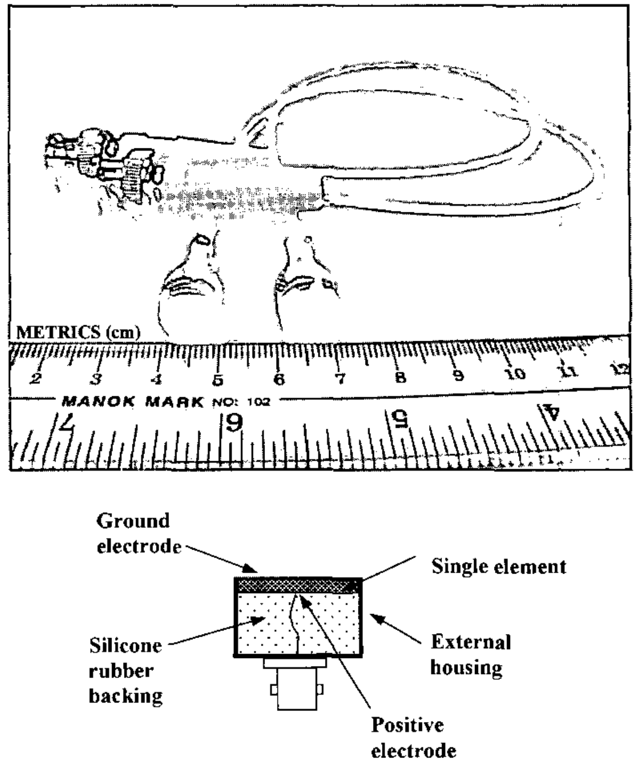

Fig. 8. Photograph of the ultrasonic transducers and a drawing showing the structure of the transducer.

IV. Properties of PMN-PT Single Crystal and PMN-PT/Epoxy 1-3 Composite Transducers

Two plane ultrasonic transducers were fabricated with the PMN-PT single crystal and the PMN-PT/epoxy 1-3 composite of 0.37 volume fraction as the driving elements, respectively. Both of the transducers have soft silicone rubber backing and have no front face matching (Fig. 8). Their performance such as the pulse-echo response and insertion loss were measured and compared. The transducer was connected to a Panametrics model 5052UA (Panametrics, Waltham, MA) ultrasonic analyzer with the setting of 4 μJ energy and 50 ohm damping. A flat stainless steel target was placed at the bottom of a water tank. The transducer was placed at the near/far field transition point T.

$$T = \frac{a^2}{4\lambda_c}, \quad (4)$$

where a is radius of transducer and λ_c is the wavelength in water at the center frequency of the transducer.

The pulse-echo waveforms were measured using an HP Infinium oscilloscope (Agilent) (Figs. 9 and 10). The fast Fourier transform of the pulse-echo waveform was stored, and the bandwidth was determined from the −6 dB points of the displayed spectrum (Figs. 11 and 12). To determine the characteristic of the transducer in its frequency domain, the following parameters from the waveforms of the single crystal transducer and the 1-3 composite transducer

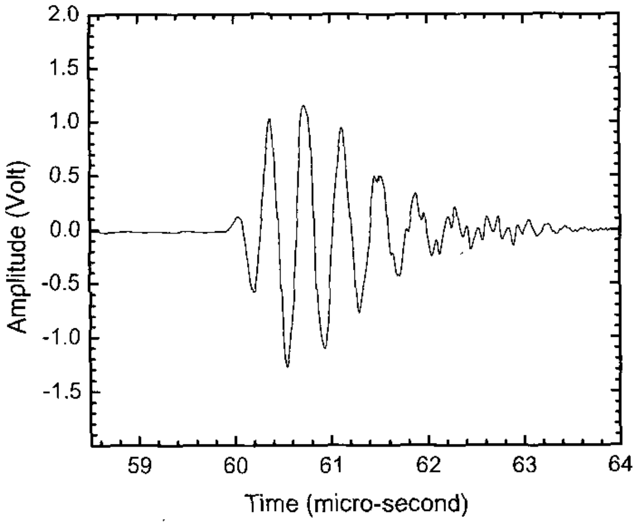

Fig. 9. Measured pulse-echo responses of the PMN-PT transducer.

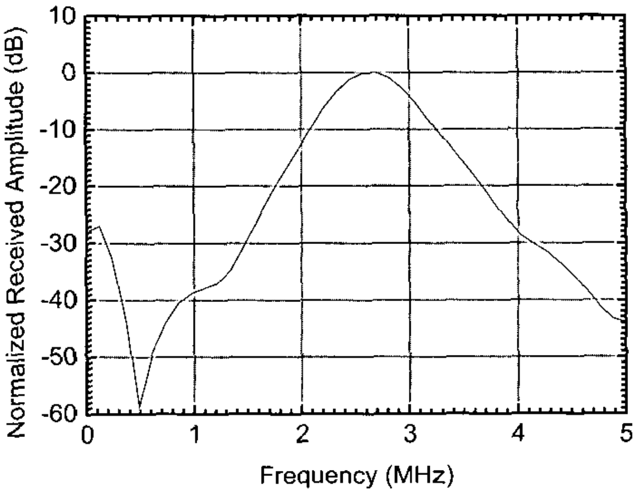

Fig. 11. Normalized received amplitude spectrum of the PMN-PT transducer.

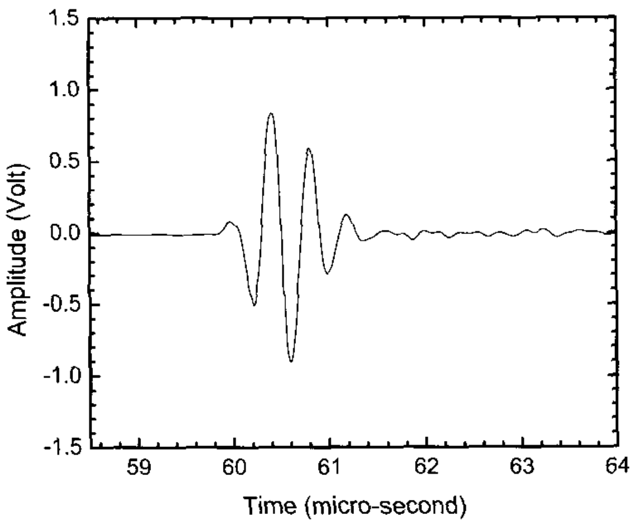

Fig. 10. Measured pulse-echo responses of the PMN-PT/epoxy 1-3 composite transducer.

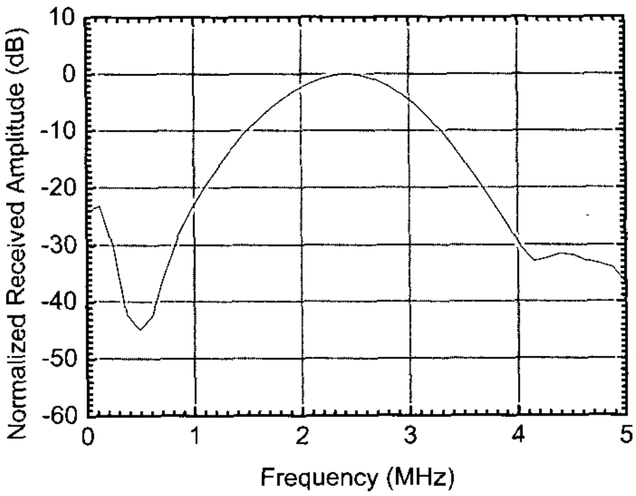

Fig. 12. Normalized received amplitude spectrum of the PMN-PT/epoxy 1-3 composite transducer.

were calculated based on the American Institute of Ultrasound in Medicine (AIUM) Standard [16]:

> Peak frequency f_p: the frequency at which the amplitude of the spectrum is the maximum. Lower and upper -6 dB frequencies (f_l and f_u): the frequencies at which the magnitude in the amplitude of the spectrum is 50% (-6 dB) of its maximum.

$$\text{Center frequency } f_c\text{: } f_c = \frac{f_l + f_u}{2}, \quad (5)$$

$$-6 \text{ dB bandwidth (percentage): } BW = \frac{f_u - f_l}{f_c} \times 100\% \quad (6)$$

The -6 dB bandwidth is a measure of the width of the frequency distribution and is an indicator of the damping factor. From their frequency spectra (Figs. 11 and 12), it was found that the center frequency of the PMN-PT single crystal transducer was about 2.64 MHz, and its -6 dB bandwidth was 31%. The PMN-PT/epoxy 1-3 composite transducer has a center frequency of 2.41 MHz and a broader -6 dB bandwidth of 61%.

The insertion loss (or the relative pulse-echo sensitivity) of the transducer is the key parameter that affects the imaging quality. It is defined as the ratio of the power of the received transducer echo power P_r to the power of the excitation pulse power P_0 delivered into the transducer. The insertion loss (IL) for each transducer was determined by:

$$IL = 10 \log \frac{P_r}{P_0} = 10 \log \frac{V_r^2/R}{V_0^2/R} = 20 \log \frac{V_r}{V_0}. \quad (7)$$

To measure the insertion loss, an HP 8116A function generator (Agilent) with 50 Ω coupling was used to generate a tone burst of 20-cycle sine wave with an amplitude V_0 of 1 V (peak-to-peak voltage) at the center frequency f_c of transducer as displayed in the HP Infinium oscilloscope with the 50 Ω coupling mode. By connecting the transducer to the function generator, the received echo voltage amplitude V_r excited from the transducer was measured by the HP Infinium oscilloscope with 1 MΩ coupling. It was found that the insertion loss for the PMN-PT single crystal transducer was about −17 dB and that of the PMN-PT/epoxy 1-3 composite transducer was about −14 dB. The IL of the PMN-PT/epoxy 1-3 composite transducer is higher than that of a PZN-PT/epoxy 1-3 composite transducer [4] with a front-face matching (with 0.5 volume fraction of PZN-PT, IL = −10 dB) and PZN-PT/epoxy composite transducer [4] also has a wider bandwidth (97%), presumably due to the effect of front-face matching.

V. Conclusions and Discussion

The PMN-PT/epoxy 1-3 composites with various volume fractions of PMN-PT were fabricated, and their electromechanical properties were measured by the resonance method. The modified parallel and series model was used to calculate the performance of the composites, and the modeling results agreed quite well with the experimental data. The composites have high electromechanical coupling coefficient of 0.8, indicating that they have good energy conversion efficiency. A single element transducer using PMN-PT/epoxy 1-3 composite was fabricated, and it had broader bandwidth compared to a PMN-PT single crystal transducer of similar structure. The use of a PMN-PT single crystal to replace conventional PZT piezoceramics is still not very viable at present because of the availability and cost of the single crystals. The batch production of high-quality homogeneous PMN-PT single crystals is still in progress. The use of these single crystals and their composites in various applications other than medical ultrasound is being developed and has attracted considerable current research interests. It is expected that more work on the applications of these single crystals will be found when the crystals are available commercially at a lower cost.

Acknowledgment

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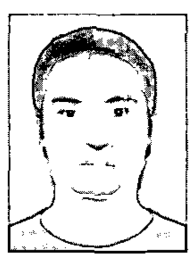

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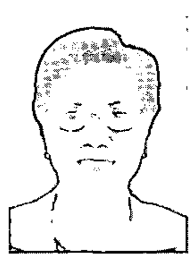

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