Samarium and Manganese-Doped Lead Titanate Ceramic Fiber/Epoxy 1–3 Composite for High-Frequency Transducer Application

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Abstract—**Samarium- (Sm) and manganese- (Mn) doped lead titanate ceramic fibers with a diameter of 35 μm were prepared using a sol-gel method. The X-ray diffraction pattern shows that the fibers have a pure perovskite structure. The 1–3 composite disks with a thickness of 31–41 μm and with ceramic volume fraction of ~0.68 have been prepared using the samarium and manganese doped lead titanate (PSmT) fibers. The resonance characteristics of the poled composite disks were measured. A focused transducer was fabricated using a concave 1–3 composite disk with nonuniform thickness in order to enhance its bandwidth. The insertion loss (IL), pulse-echo response and frequency spectrum of the composite transducer were measured. The center frequency of the transducer was ~31 MHz with a −3 dB bandwidth of ~123% and a low IL of 29.3 dB.**

I. Introduction

THE need to increase the resolution of ultrasonic imaging (e.g., in the imaging of arteries, in dermatology, and in ophthalmology) has called for the use of high-frequency (>20 MHz) ultrasonic transducers [1]–[4]. Piezoelectric ceramic fiber/epoxy 1–3 composites consisting of piezoelectric ceramic fibers embedded in an epoxy matrix are excellent materials for fabricating high frequency transducers as they have high thickness electromechanical coupling coefficients and low planar coupling coefficients [5]–[14]. The ceramic/polymer composites are not as brittle as the thin piezoceramic plate and can easily be shaped into a lens. Very fine scale ceramic fibers can be used, and the volume fraction ϕ of the ceramic fiber can be varied to adjust the electrical impedance of the transducer. Doped-lead zirconate titanate (PZT) has high d_{33} and high thickness electromechanical coupling coefficient k_t; but its planar mode electromechanical coupling coefficient k_p is also high. Hence, if the thickness of the PZT fiber inside the 1–3 composite is close to its diameter, the lateral and thickness resonance modes will couple and pure thickness mode resonance cannot be generated [12], [15]. So when PZT ceramic is used to fabricate 1–3 composites, the aspect (diameter/length) ratio of a single PZT fiber in the 1–3 composite should be at least <0.5. In other words, the length of the fiber should be larger than twice the diameter. Moreover, for high-frequency, single element transducers have a large area compared to their thickness. The high relative permittivity (1500~4000) of the PZT material results in an electrical impedance ($Z = 1/\omega C$, where ω is the angular frequency and C is the capacitance) that is substantially lower than 50 Ω. Samarium- and manganese-modified lead titanate [$Pb_{0.85}Sm_{0.1}Ti_{0.98}Mn_{0.02}O_3$ (PSmT)] ceramics exhibit large electromechanical anisotropy [16]–[18] and will be good candidates for high-frequency 1–3 composite ultrasonic transducers because of their very low relative permittivity (~185) and low planar coupling coefficient ($k_p = 0.05$). When PSmT fibers are used to fabricate 1–3 composites, coupling between the planar and thickness mode can be alleviated, and the aspect ratio of the fiber can be much closer to 1, which facilitates the fabrication of high frequency transducers.

Sol-gel methods have been used to prepare PZT and calcium-doped PT thin films and coatings in microdevice fabrications [19], [20]. The materials prepared using the sol-gel methods have good homogeneity, and it is relatively easy to vary the chemical composition. In recent years, there are reports on the preparation of PZT and PLZT ceramic fibers by the sol-gel methods [21]–[26]. Compared with Ca modified PT (another doped PT composition that has high electromechanical anisotropy) in which small quantities of three to four different dopants have to be added, PSmT has fewer components. Hence, it is easier to prepare PSmT fibers by using the sol-gel method.

II. Experiment

The precursors used in this study were lead acetate trihydrate, titanium n-butoxide (or titanium iso-propoxide), manganese acetate dihydrate, and samarium acetate hydrate (from Strem Chemicals, Newburyport, MA).

A. Preparation of PSmT Ceramic Fibers

The nominal composition of the samarium- and manganese-doped lead titanate ceramic fiber is $Pb_{0.85}Sm_{0.1}Ti_{0.98}Mn_{0.02}O_3$ (PSmT). The process of preparing the sol-gel solution and fabricating the ceramic fibers is shown in Fig. 1. Due to the large area of the fibers, 2% excess lead was added in preparing the solution to compensate for the lead loss in the sintering process. Gel fibers were

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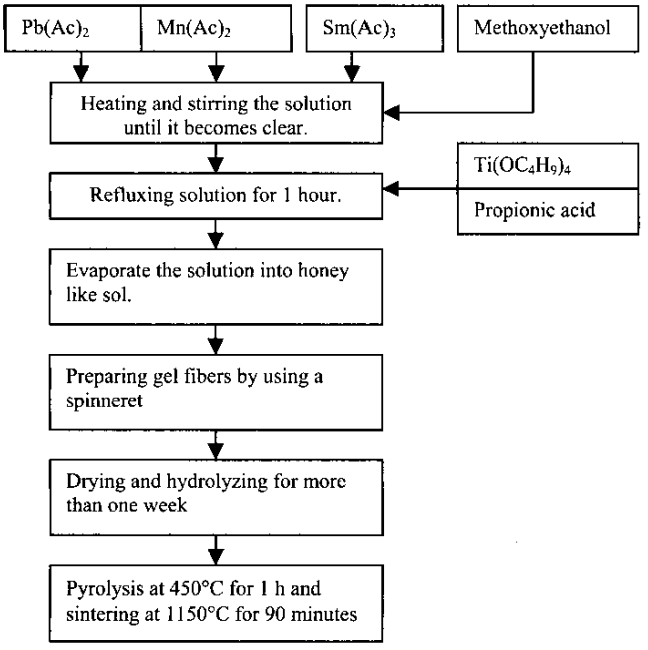

Fig. 1. The processes of preparing PSmT ceramic fibers.

spun from spinnerets with pinholes of various diameters using a spinning machine (OneShot III, from Alex James & Assoc., Inc., Greenville) [20], [21]. As the gel fibers have 40–50% shrinkage during subsequent heat treatments, ceramic fibers with 35–100-μm diameter can be prepared by using different spinnerets with 100–250-μm pinholes and by changing the speed of rotation of the spindle.

The gel fibers were tied together in a bundle and were hydrolyzed and dried at room temperature for more than 1 week. Then they were dried at 60–80°C for 8–12 hours. The dried fibers were placed on top of a layer of PSmT ceramic powder, which has the same composition as the fibers, on an alumina plate. A spoonful of carbon black was scattered around the fiber bundle to prevent fiber cracking in the pyrolysis. They were covered with an alumina crucible, pyrolyzed at 400°C for 1 hour and at 550°C for 1 hour. The pyrolyzed fibers were calcinated at 850°C for ~2 hours and then sintered at 1150°C for 1.5 hours to form the PSmT ceramic fibers. From the scanning electron micrograph of the sintered PSmT fiber (not shown here), it is seen that the grains in the fiber are well grown, and it is relatively dense. The sintering temperature and time were selected to control the grain size of the fiber. The ceramic grain diameter normally should be less than 1/10 of the fiber diameter to give the fiber appropriate strength.

B. Fabrication of PSmT Ceramic Fiber/Epoxy 1–3 Composites

The sintered bundle of PSmT ceramic fibers was inserted into a plastic tube with a diameter of ~6 mm. The tube was then filled with low viscosity epoxy [Spurr hardness B, vinylcyclohexene dioxide (VCD)/diglycidyl ether of polypropyleneglycol (D.E.R 736)/noneenyl succinic anhydride (NSA)/dimethylaminoethanol (DMAE) = 10:4:26:0.4 in weight ratio]. The longitudinal and transverse wave velocity v_L and v_T of the Spurr epoxy (hardness B) were measured using an ultrasonic immersion technique [27]; other stiffness constants were then calculated using the measured v_L and v_T. The relevant parameters are listed in Table I. After evacuation to eliminate the trapped gas, in order to prepare a composite with a high volume fraction of ceramics, the tube was tightly wrapped with a rubber tape so the fibers were packed together closely. The composite rod then was heated at 80°C for 8 hours to fully cure the epoxy before it was cut into disks with a thickness of ~200 μm using a diamond saw. They then were ground and polished to the desired thickness. After chromium/gold layers were evaporated on both sides of the disks as electrodes, the disks were poled under an electric field of 4.5 kV/mm at 115–120°C for 15 minutes. In order to fabricate a concave disk [28], a composite disk was adhered to a convex lens, then ground and polished carefully with fine alumina powder to the desired thickness. Then chromium/gold was evaporated on the polished sides of the disk as the electrodes. The resonance characteristics of the disks were measured using an impedance analyzer HP4294A (Agilent, Palo Alto, CA). The ceramic volume fraction ϕ of the composite disk was measured by integrating the surface area of ceramic fibers under an electron scanning microscope (SEM), and it was found to be ~0.68.

TABLE I
MATERIAL PARAMETERS OF THE SPURR EPOXY AS MEASURED BY THE ULTRASONIC IMMERSION TECHNIQUE [27].

Properties	Spurr epoxy
Relative permittivity ε (at 1 kHz)	3.27
Density ρ (kg/m^3)	1102
v_L (m/s)	2209
v_T (m/s)	978
Stiffness $c_{11} = c_{22} = c_{33}$ (GPa)	5.377
Stiffness $c_{44} = c_{55} = c_{66}$ (GPa)	1.054
Stiffness $c_{12} = c_{21} = c_{23} = c_{32} = c_{13} = c_{31}$ (GPa)	3.269
Compliance $s_{11} = s_{22} = s_{33}$ (pm^2/N)	344.2
Compliance $s_{44} = s_{55} = s_{66}$ (pm^2/N)	948.8
Compliance $s_{12} = s_{21} = s_{23} = s_{32} = s_{13} = s_{31}$ (pm^2/N)	130.2
Young's modulus, Y (GPa)	2.905

C. Fabrication of High-Frequency Transducer

A stainless steel tube was screwed onto a standard male UHF connector to form a transducer housing (Fig. 2). A piece of fine copper wire with 50-μm diameter was soldered onto the center electrode of the connector, then connected to the bottom electrode of the composite. The gap between the screw was sealed with epoxy. The steel tube was filled with silicone rubber (noncorrosive silicone rubber, RS Components, Corby, Northants, UK). On the top part of the tube adjacent to the composite disk, some foam rubber beads were incorporated to form a porous back-

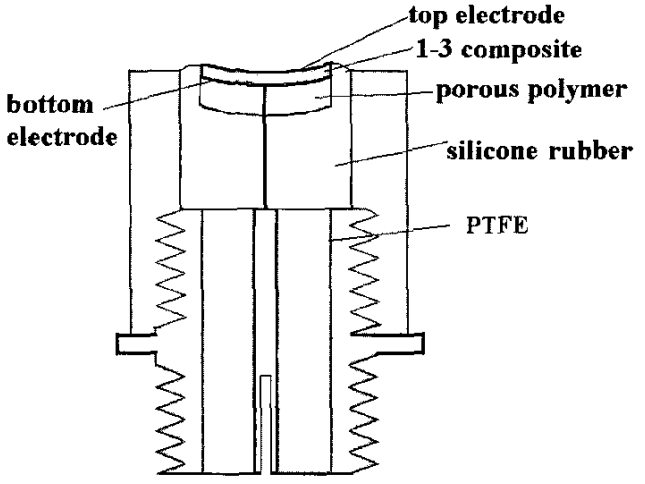

Fig. 2. Diagram of a 1–3 composite transducer.

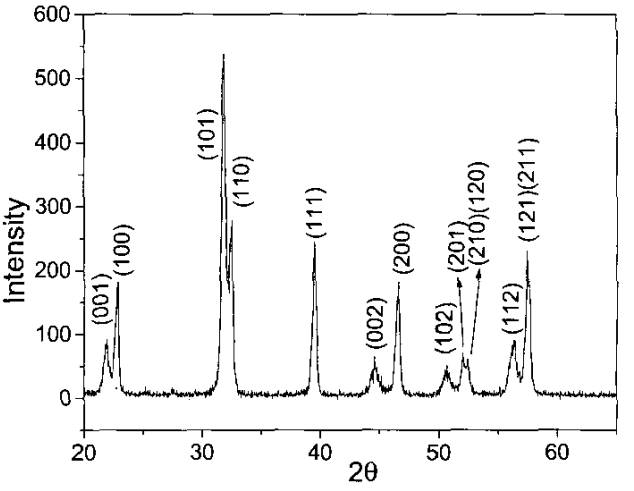

Fig. 3. X-ray diffraction pattern of the PSmT ceramic fiber.

ing with a thickness of ~1 mm. This porous backing has a low acoustic impedance and can reduce the ultrasonic wave propagating into the backing. A PSmT 1–3 composite disk with a diameter of 5 mm was attached to the porous backing. Before the backing was fully cured, a 30-mm diameter steel ball was used to press on the top to form a concave surface. After the backing was cured and the top electrode connected to the steel housing, a focused transducer was made.

III. Characterization of the 1–3 Composite Transducers

The transducer was placed in a water tank with a planar stainless steel target at the focus to obtain a maximized echo. A step electrical pulse was produced from a Panametrics 5900 (Panametrics, Waltham, MA) pulser-receiver to drive the transducer. The spatial waveform of the pulse/echo response was recorded by a digital oscilloscope (HP Infinium 54820A, Agilent). The frequency spectrum was obtained by using the fast Fourier transform (FFT) function of the oscilloscope. In the frequency spectrum, the maximum frequency f_{max}, f_l, and f_u (f_l and f_u are frequencies at the −6 dB or half amplitude points) can be obtained. The center frequency (f_c) and percentage bandwidth (BW) can be calculated by the following equations:

$$f_c = \frac{f_u + f_l}{2}. \tag{1}$$

$$\mathrm{BW} = \frac{f_u - f_l}{f_c} \times 100\%. \tag{2}$$

The IL (or the relative pulse-echo sensitivity) of the transducers is defined as the ratio of the received transducer echo power P_r versus the pulse excitation power P_t, which is expressed in decibels as:

$$\mathrm{IL} = 10 \log \left(\frac{P_r}{P_t} \right) \text{ dB}. \tag{3}$$

To measure the IL, an HP8116A (Agilent) pulsed/function generator was used to generate a toneburst of 20 cycles with an amplitude of 1 V peak-to-peak at the center frequency of the transducer. The transducer was immersed in water facing a flat stainless steel target placed at the focus. Amplitude of the echo P_r was measured and as $P_t = 1$ V, IL can be calculated by (3).

IV. Results and Discussion

Fig. 3 shows the X-ray diffraction pattern of the PSmT fiber sintered at 1150°C for 1.5 hours. The (002) and (200) peaks are clearly separated showing that the PSmT ceramic fiber has a tetragonal crystal phase. The parameters of the unit cell are a = 3.8991 Å and c = 4.0543 Å. According to the formula weight of $Pb_{0.85}Sm_{0.1}Ti_{0.98}Mn_{0.02}O_3$, the theoretical density of the PSmT ceramic fiber is 7730 kg/m^3. The density of the fiber was measured using the Archimedes principle and was found to be 7650 kg/m^3, showing that some pinholes may still exist in the interior of the fibers.

Fig. 4 shows the SEM micrograph of a PSmT ceramic fiber/epoxy 1–3 composite. The PSmT fibers, prepared by using a 100-μm pinhole, were used in fabricating the 1–3 composites. The diameter of the ceramic fibers determined from an enlarged SEM micrograph is ~35 μm. The ceramic volume fraction ϕ is ~0.68, as estimated by integrating the surface area of the ceramic fibers.

From the impedance and phase spectra of a 34-μm thick 1–3 composite disk with a diameter of 4.5 mm, only a weak lateral resonance peak can be observed in the phase spectrum (Fig. 5), which indicates that the composite has very weak planar resonance. Fig. 6 shows the impedance and phase spectra of this sample in the 10–100 MHz frequency range, and a clean thickness mode resonance ($k_t \sim 0.51$) is observed at ~50 MHz; even the fiber has a diameter/thickness ratio of ~1 indicating that the coupling be-

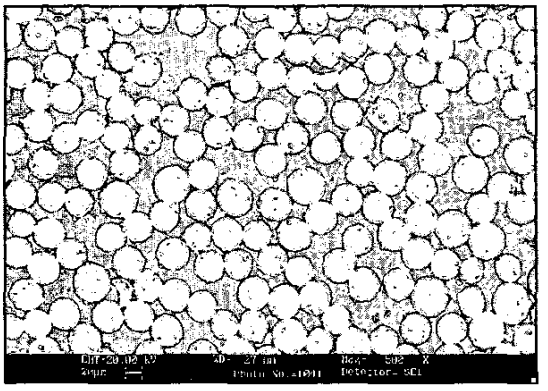

Fig. 4. SEM micrograph of PSmT ceramic fiber/epoxy 1–3 composite with $\phi = 0.68$.

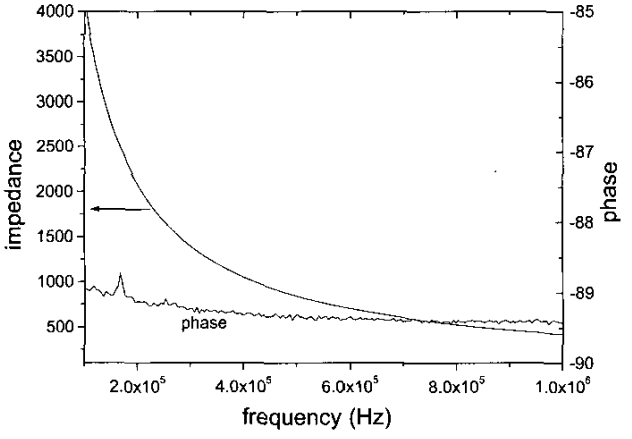

Fig. 5. The impedance spectra of a 1–3 composite disk (thickness = 31 μm) in the low-frequency region. The upper solid line represents the impedance, the lower line represents the phase.

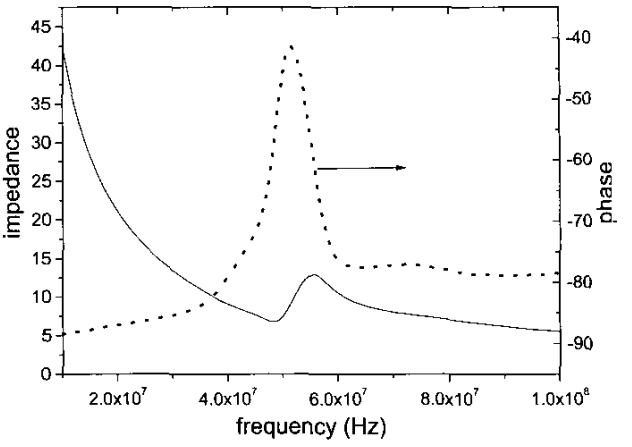

Fig. 6. The impedance spectra of a 1–3 composite disk (thickness = 31 μm) in the high-frequency region. The solid and dotted lines represent the impedance and phase, respectively.

TABLE II
MEASURED PARAMETERS OF THE PSMT FIBER/EPOXY 1–3 COMPOSITE WITH 35-μM DIAMETER FIBERS AND $\phi = 0.68$.

Property	PSmT fiber/epoxy 1–3 composite (35-μm fiber, $\phi = 0.68$)
Relative permittivity ε (at 1 kHz)	120
Density (kg/m^3)	5480
k_t	0.51
d_{33} (pC/N)	48
$\tan\delta$	0.013
N_t (mHz)	1695

tween the planar and thickness modes in the ceramic fiber has been alleviated by using the anisotropic PSmT ceramics. According to the IEEE standards [29], other parameters of the PSmT 1–3 composites were measured and calculated. The results listed in Table II were obtained by averaging over ~10 composite samples.

Fig. 7 shows the impedance and phase spectra of a 5-mm diameter disk. This concave composite disk has a thickness (t_{center}) of 35 μm at the center and t_{edge} of 41 μm at the edge. The value of $\Delta f(\Delta f = f_a - f_r)$ of the concave disk is larger than that of a uniform disk and $k_t \sim 0.56$. The value of k_t increases from 0.51 to 0.56 as $\Delta t = t_{edge} - t_{center}$ varies from 0 to 6 μm. However, when Δt was increased further, both the impedance and the phase peaks split into two or more peaks. This is because each fiber has its own thickness resonance, and the thickness resonance of the 1–3 composite is a resultant of the collective resonance of all the fibers. If the length of the fibers was nearly the same, their resonances have similar values, which result in a sharper thickness resonance. If the length of the pillars are slightly different, their res-

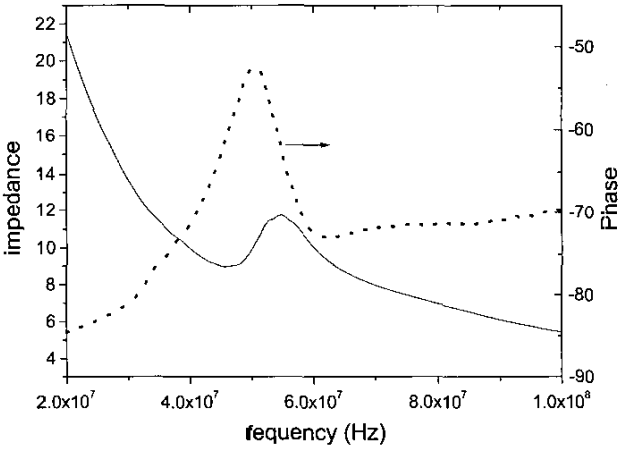

Fig. 7. The impedance and phase spectra of a nonuniform concave 1–3 composite disk ($t_{center} = 35$ μm, $t_{edge} = 41$ μm). The solid and dotted lines represent the impedance and phase, respectively.

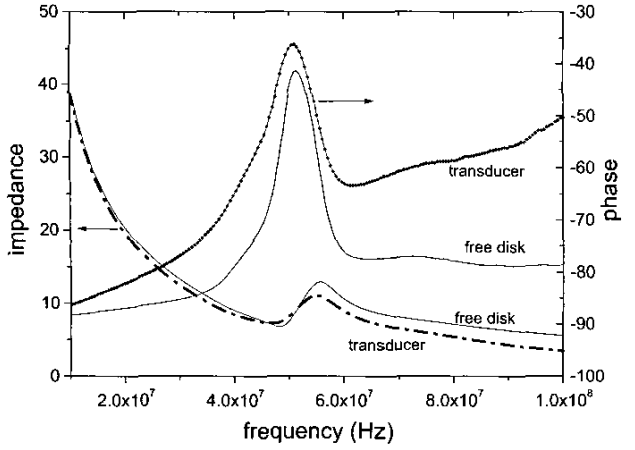

Fig. 8. Comparison between the spectra of a nonuniform disk (t_{center} = 32 μm, t_{edge} = 35 μm) and the transducer made from this disk.

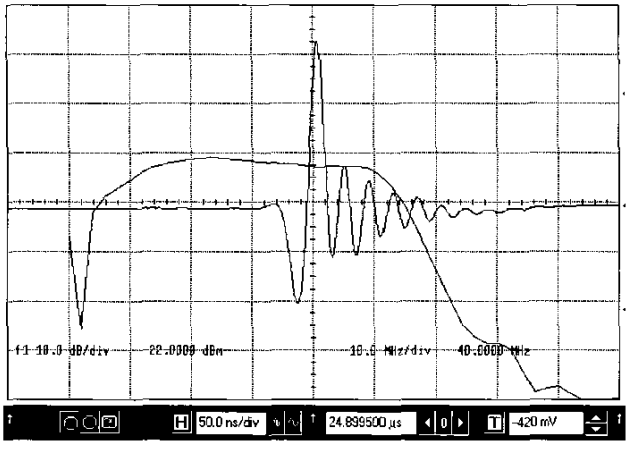

Fig. 9. The pulse/echo response and frequency spectrum of the 1–3 composite transducer with nonuniform thickness.

onance peaks appear at slightly different frequencies, and a broader resonance peak with larger Δf will be observed. Using this observed phenomenon, a broadband transducer may be fabricated [30].

Fig. 8 shows the impedance and phase spectra of a concave 1–3 composite disk and the transducer made from a disk with a thickness of 32 μm at the center and 35 μm at the edge. It is seen that even a light porous polymer backing was used, the peak impedance of the transducer is lower than that of the free disk, indicating that the backing has absorbed some ultrasonic energy. The resonance frequency has shifted to a lower frequency because the back surface of the disk is slightly constrained by the backing.

Fig. 9 shows the spatial pulse/echo response of this focused 1–3 composite transducer. The measuring conditions were: repetition rate = 10 kHz, energy = 2 μJ, damping = 50 Ω, attenuation = 25 dB, gain = 40 dB, low-pass filter = 200 MHz, high-pass filter = 1.0 MHz. The uncorrected frequency spectrum of the transducer was obtained by using the FFT option built-in to the HP Infinium 54820A (Agilent) oscilloscope and shown in Fig. 9. If the data were downloaded and FFT was performed after patching zeros, the bandwidth decreased by ~15%. The composite transducer has a center frequency at ~31 MHz and a broad bandwidth of ~123% at −3 dB (~105% after patching zeros) and ~139% at −6 dB (~118% after patching zeros), which is higher than the reported values for PZT fiber/epoxy 1–3 composite transducers. The IL is 29.3 dB, which also is better that the reported IL of PZT fiber/epoxy transducers [31], [32], presumably due to the high volume fraction of PSmT fiber ϕ used in the present work. In the present work, $\phi \sim 68$ while in [31] and [32] $\phi = 0.21$ (IL = 47 dB) and 0.45 (34.4 dB), respectively. The focal length of the transducer was ~17.5 mm, which was determined by the diameter of the steel ball used to form the curvature in the composite disk; and it can be varied if a steel ball of a different diameter is used. For such a broad bandwidth transducer, the ringdown time should be very short. However, several wavelengths were observed in the pulse-echo time domain waveform. This may be due to the fact that the fibers in the composites have slightly different length and resonance frequencies, and they are not equidistance from the reflector. Hence, echoes from different parts of the transducer occur at slightly different time scale and form several cycles in the resultant time domain waveform. If we examine these several cycles in the waveform carefully, we can see that the first cycle has a longer period and, hence, is of lower frequency compared to the subsequent ones. It is expected that the performance of the transducer can be further enhanced if a suitable front-face matching layer could be used.

V. Conclusions

The PSmT ceramic fibers with diameter ~35 μm have been fabricated using a sol-gel method. The ceramic fibers were found to have a perovskite crystal phase and good piezoelectric properties. The PSmT ceramic fiber/epoxy 1–3 composites with 0.68 ceramic volume fraction were fabricated. The impedance and phase spectra showed that the lateral resonance peaks almost disappeared, and a clean thickness resonance peak can be observed at 31 MHz with a fiber aspect ratio close to 1, which shows the advantage of using the anisotropic PSmT fibers. By using a concave disk with nonuniform thickness, the resonance peak can be broadened, which results in a higher k_t, and a broadband transducer can be fabricated. Broad bandwidths of ~123% at −3 dB (~105% after patching zeros) and 139% at −6 dB (~118% after patching zeros) and a low IL of 29.3 dB were obtained.

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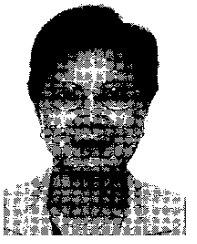

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