Single Crystal Piezoelectric Composite Transducers for Ultrasound NDE Applications

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ABSTRACT

Single crystal piezoelectric composite transducers including 75 MHz PC-MUT (piezoelectric composite micromachined ultrasound transducers), diced 10 MHz and 15 MHz 1-3 composite transducers were successfully demonstrated with broad bandwidth and high sensitivity. In this paper, the design, fabrication and characterization of composite transducers are reported. C-scan experiments for SiC ceramic samples were performed using these composite transducers as well as some commercial NDE transducers. The results suggest that significant improvements in resolution and penetration depth can be achieved in C-scan NDE imaging using single crystal composite broadband transducers.

Keywords: single crystal piezoelectrics, piezoelectric composites, NDE, NDT, ultrasound transducers.

1. INTRODUCTION

Ultrasound can be used to detect internal defects and anomalies nondestructively at resolutions that cannot be duplicated by other types of test systems [1]. Pulse-echo techniques where transmission and receiving is done with the same transducer, is the most common ultrasound NDE imaging method. Determination of the amplitude of ultrasound reflected back to the transducer at an internal interface such as a defect, voids, delamination, etc. is used to detect surface and subsurface damage in ceramic materials. Because of the very small size of relevant defects in high quality ceramic components, high frequency acoustic microscopy is generally used in such inspections. In acoustic microscopy a spherical lens is usually used to focus the ultrasound beam to a spot, the transverse resolution, axial resolution and depth of focus are critical to obtaining high resolution and large imaging volumes. High resolution is needed to observe the defects of interest and large imaging volumes are necessary to minimize the inspection time of large components. According to traditional ultrasound theory, short wavelength (high frequency) and short pulse-width of the wave (high frequency, broadband) are desired for ultrasound imaging with high axial and lateral resolutions in applications including dermatology, ophthalmology, intravascular imaging, laproscopy and NDE imaging [1-3]. In recent years a variety of methods have been investigated for constructing high frequency broadband ultrasound transducers. Piezoelectric composites enable a more efficient utilization of the piezoelectric response due to the reduction in internal clamping within piezoelectrics; specifically the posts formed in 1-3 composites allow for the full longitudinal, lengthextensional ("33") mode to operate. Transducers based on this architecture exhibit high bandwidth, high sensitivity, and good array properties (low inter-element cross talk, low side-lobe levels) [4,5]. Conventional composite transducers operated below 20 MHz are constructed by dicing piezoelectric ceramics and then backfilling the saw cuts (kerfs) with epoxy to form composite structures. However, this so-called dice-and-fill method of transducer fabrication cannot be used to make transducers that operate much above 20 MHz. Alternative methods include use of PVDF piezoelectric polymer, PbTiO₃ ceramic, LiNbO₃ crystals, or ZnO thin films. All these approaches make use of materials with considerably lower piezoelectric properties than PZT-polymer composites. Alternative composite fabrication methods

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Nondestructive Characterization for Composite Materials, Aerospace Engineering, Civil Infrastructure, and Homeland Security 2008, edited by Peter J. Shull, H. Felix Wu, Aaron A. Diaz, Dietmar W. Vogel, Proc. of SPIE Vol. 6934, 69340D, (2008) · 0277-786X/08/\$18 · doi: 10.1117/12.776186 based on stacked plates [6] or tape casting technology [7] have shown promise in recent years, but the development of single crystal piezoelectrics based on $(1-x)Pb(Mg_{1/3}Nb_{2/3})O_3$ -xPbTiO₃(PMN-PT) provides a means of advancing the performance of high frequency transducers far beyond the capability of conventional ceramic devices. NDE imaging can benefit greatly from high frequency ultrasonic imaging using transducers based on piezoelectric single crystals such as PMN-PT [8,9]. The high electromechanical coupling ($k_{33} > 90\%$) and large piezoelectric charge coefficient ($d_{33} > 2000$ pC/N) result in transducers that exhibit more bandwidth and sensitivity than their ceramic (PZT) counterparts. The combination provides a means of both improving resolution (high frequency content) and depth of imaging over existing devices. As stated before, traditional dicing techniques (precision saws with < 20 µm thick diamond blades) are limited to 20 MHz composites, however, the structure of PMN-PT single crystal provides a distinct advantage in that it enables the use of a broad range of micromachining methods developed for integrated circuit fabrication and micro-electromechanical systems (MEMS). TRS developed a process capable of micro-machining PMN-PT single crystal into 20 - > 100 MHz composites using Deep Reactive Ion Etching (DRIE). Both 1-3 and 2-2 composite structures with feature sizes appropriate for operation up to 75 MHz were fabricated for high frequency transducers [10,11].

In this paper, fabrication and testing of single crystal piezoelectric composite transducers are presented for NDT ultrasound. Both the dice-and-fill technique and micromachining technology were used for single crystal composite preparation. Impedance and phase spectrum of composites were recorded using an impedance analyzer. Pulse-echo tests were performed to characterize composite transducers. The prototyped composite transducers were then applied in c-scan experiments for SiC samples.

2. EXPERIMENTAL DESIGN

2.1 Single crystal 1-3 composite transducers

Single crystal 1-3 composites with frequency of 10 MHz, 15 MHz, 60 MHz, and 75 MHz were designed, fabricated and characterized. The 10 MHz and 15 MHz 1-3 composites were fabricated using a dice-and-fill technique, as shown in Figure 1 (a). The 75 MHz composites were fabricated using the recently developed PC-MUT technology (Figure 1(b)) [11]. The details on the composite design, fabrication and characterization can be found in our previous papers [11,12]. Transducers were prototyped using these composites and tested to determine the sensitivity and bandwidth.



Figure 1. Composite transducers fabrication processes. (a) dice-and-fill process. (b) PC-MUT process.

2.1.1 Diced single crystal 1-3 composite transducers

A. 10 MHz and 15 MHz 1-3 composites

For 10 MHz and 15 MHz composite fabrications, PMN-PT single crystal plates with dimension of 15 mm x 15 mm x 1 mm were diced in X and Y axis first, followed by kerf filling, lapping and electroding (Cr/Au). The final thickness for 10 MHz composite is about 0.125 mm, and the thickness for the 15 MHz composites is about 0.1 mm. The composites

were poled at 10 KV/cm at room temperature, followed by characterization. Dielectric constant and dielectric loss were measured using an impedance analyzer (HP4194A) at a frequency of 1 KHz. The impedance and phase spectrum were recorded using the impedance analyzer and the electromechanical coupling coefficients were then calculated using the IEEE standard for the k_{33} coupling.

B. 10 MHz and 15 MHz composite transducers

Figure 2 shows the schematic cross view of a 10 MHz transducer. A $\phi 6$ mm x 0.125 mm 10 MHz PMN-PT 1-3 composites disc was prepared with Cr/Au electrode on both top and bottom surfaces. A matching layer was cured, lapped to thickness (~50 microns), and bonded to the front of the composite disc using epoxy. This provided a very thin bond layer. A backing layer was bonded to the back of the device. The transducer was then core drilled to achieve the final 6 mm diameter. A wire was connected to the backing to achieve the active connection, and the transducer was then potted into a housing using a potting compound. A Cr/Au layer was sputtered onto the transducer face to achieve the ground connection to the housing, and a UHF connector was connected to the back of the device. Fabrication of the 15 MHz transducer was similar to the 10 MHz device, except that the transducer was a focused one. The transducers were re-poled subsequent to fabrication, to insure the device was fully poled. The 10 MHz and 15 MHz transducers were subsequently put into water tank for pulse-echo tests using a Panametrics/Olympus 5052PR pulser-receiver.



Figure 2. 10 MHz PMN-PT 1-3 composite transducer assembly.

2.1.2 PC-MUT

A. 75 MHz Composites

PMN-PT single crystal discs with dimension of ϕ 15 mm x 0.6 mm were lapped and polished on one side for lithography. The following processes, including electroplating, deep reactive ion etching, kerf filling, lapping and electroding, were then performed to fabricate the 75 MHz composites. After electroding, PC-MUT composites were poled at 10 KV/cm at room temperature, and the characterization was then performed following the same procedure as the 10 MHz composites. The capacitance, dielectric loss and resonance frequencies were measured using the HP impedance analyzer, and the electromechanical coupling coefficients were then calculated.

<u>B. 75 MHz PC-MUT</u>

After the wafers were micromachined, the etched kerfs were filled with epoxy, and lapping was then performed on one side. The first lapped side of the composite was sputtered with a Cr/Au electrode, and the silver epoxy layer was then cast onto the composite and lapped to a thickness of 500 microns. The plate was next flipped, and the composite was lapped to a final thickness of about 20 microns, followed by Cr/Au electroding. Small squares were diced from the wafer, and a 0.5-0.6 mm square was used to fabricate the device (similar to the structure shown in Figure 2). After potting into the brass housing, the face was sputtered with Cr/Au to achieve the ground connection, and an SMA connector was attached for the interconnection. The transducer was poled at 12 kV/cm at room temperature. The pulse-echo response of the 75 MHz transducer was tested using a Panametrics/Olympus 5900PR pulser-receiver and a steel plate echo source. The pulse-echo result obtained from the 75 MHz PC-MUT was compared with the one from a commercial 75 MHz focused transducer .

2.2 C-Scan imaging experiments

TRS 10 MHz, 15 MHz and 75 MHz immersion transducers were tested for sensitivity and defect resolution in solid ceramic materials (SiC and alumina). The capabilities of these transducers were also compared with those commercially available transducers. C-scans were set up using the SecondWave Studio program at Rutgers University. This program also served as a digital oscilloscope. A JSR DPR500 pulser/receiver was used in conjunction with an RP-H2 remote

pulser that was connected to the transducer using a microdot connection. The pulser and receiver settings were controlled digitally using a digital JSR control panel. This panel allowed for control of pulse energy, damping, and receiver gain. The TRS transducers were first set up in a water tank above a tile of sintered alumina (Al_2O_3) to see its interaction with the sample. The waveforms recorded can be used as for a direct sensitivity comparison between TRS transducers and commercial ones. The pulse reflection from the top surface and the bottom surface of the tile were recorded to determine the time of flight from tile surfaces to the transducer surface and the amplitude, which can then be used to form C-Scan images [14].

3. EXPERIMENTAL RESULTS

3.1 Single crystal 1-3 composite transducers

Table 1 below shows approximate kerf widths corresponding to the frequencies of interest for the composite transducers prototyped in this work. Single crystal 1-3 composite transducers with frequency of 10 MHz, 15 MHz, and 75 MHz were prototyped and characterized. Figure 3 shows the picture of assembled transducers.

Table 1. Kerl and post dimensions for different frequency composites.								
1-3 composite frequency (MHz)	Kerf width (µm)	Post Width (µm)	Approximate thickness (µm)					
10	21	63	140					
15	14	42	95					
75	2.9	9	18					

Table 1. Kerf and post dimensions for different frequency composites.



Figure 3. Three transducers developed in program: 75 MHz, 10 MHz and 15 MHz designs, from left to right. Electrical connection is achieved with an SMA connector for the 75 MHz device, and UHF connectors for the others.

3.1.1 Diced composite transducers

A. 10 MHz and 15 MHz composites fabrication and characterization

Figure 4 shows the photograph pictures of 10 MHz and 15 MHz composites. Figure 5 shows the impedance and phase spectrum of the fabricated single crystal 1-3 composites with frequency of 10 MHz and 15 MHz. Table 2 shows the measured dielectric constant, dielectric loss and the electromechanical coupling coefficients of the fabricated 10 MHz and 15 MHz composites. High electromechanical coupling coefficients were observed, which is essential to fabricating transducers with high sensitivity and broad bandwidth.



Figure 4. Photograph of 10 MHz (left) and 15 MHz (right) single crystal 1-3 composites fabricated using dice-and-fill technique.



Figure 5. Impedance and phase spectrum of single crystal 1-3 composites. The 10 MHz composite dimension is 3.99 mm x 3.72 mm x 0.124 mm, and the 15 MHz composite dimension is 3.62 mm x 3.47 mm x 0.101 mm.

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	Frequency	Dielectric constant	Dielectric constant	Dielectric loss	Coupling coefficient					
		$(\mathbf{K}^{\mathrm{T}})$	(K^{S})							
	10 MHz	2135	527	0.016	0.79					
	15 MHz	1503	383	0.012	0.76					

Table 2. Properties of 10MHz and 15 MHz composites.

B. 10 MHz and 15 MHz 1-3 composite transducers

The 10 MHz transducers prototyped were put into a water tank for pulse-echo tests using Panametrics/Olympus 5052PR pulser-receiver, and Figure 6(a) shows the typical pulse-echo spectrum for 10 MHz transducers fabricated. The bandwidth of the tested 10 MHz transducers was about 70-91%, the loop sensitivity is about -28 dB, the -20 dB pulse width is about 0.28 μ s. With the short pulse, higher sensitivity, these 10 MHz transducers are promising for SiC C-scan imaging. Pulse-echo test of a 15 MHz focused transducer was also performed using the same setup. Figure 6 (b) shows the pulse-echo spectrum of a 15 MHz transducer. The centre frequency is about 12.1 MHz, a little bit lower than designed, which is caused by the thicker composites (100 μ m instead of ~ 95 μ m as designed). The focal length was measured to be ~ 34.5 mm, the sensitivity is about -13 dB. The bandwidth is about 131%, though the waveform shape needs to be further improved. The -20 dB pulse width of 15 MHz transducer is about 0.20 μ s.



Figure 6. Pulse-echo spectrum of single crystal 1-3 composite transducers. (a) 10 MHz transducer; (b) 15 MHz transducer.

3.1.2 PC-MUT transducers

A. PC-MUT composites

Figure 7 shows an SEM picture of etched PMN-PT single crystal posts for 75 MHz composites. The micro-posts with high aspect ratio were achieved, which is important to fabricate high frequency 1-3 composites. Figure 8 shows the photograph pictures of both sides of 75 MHz 1-3 composites with Cr/Au electrode. Fine kerf width control was observed. Figure 9 shows the impedance and phase spectrum of micromachined 75 MHz composites. For the first time, > 40 MHz

PMN-PT single crystal 1-3 composites were successfully fabricated showing strong and clear resonances. The major properties of the high frequency 1-3 composites are presented in Table 3.



Figure 7. SEM picture of etched PMN-PT posts for PCMUT composites. The micro-posts are about 8 μ m wide and > 40 μ m long.



Figure 8. Photograph pictures of PC-MUT composites (both top (left) and bottom (right) surfaces) (75 MHz) with kerf $< 3 \mu m$.



Figure 9. Impedance and phase spectrum for 60 MHz (a) and 75 MHz (b) composites.

Samples	f _r (MHz)	f _a (MHz)	k _{eff}	Kerf (µm)	Thickness (µm)	Dielectric loss
75 MHz sample	60.1	78.1	0.67	< 3	~ 20	0.06

B. 75 MHz PC-MUT transducers

The pulse-echo responses of 75 MHz transducers were tested using a Panametrics/Olympus 5900PR pulser-receiver, with 15 dB total gain and the lowest energy setting. The echo target was a stainless steel plate placed in DI water at room temperature. A Tektronix 644B oscilloscope was used to digitize the echo signal, and spectral analysis was done with MATLABTM. A commercial 75 MHz transducer (1/4" aperture, $\frac{1}{2}$ " focal length) was tested to compare the

performance with the single crystal device. The commercial transducer used a quartz delay line for focusing the acoustic beam. All testing conditions were maintained between the two transducers. The comparison in acoustic signals is shown in Figure 10. The most noticeable difference in signals is the amplitude, the PMN-PT composite transducer produces a much stronger signal than the commercial device. As can be seen in the spectrum, which is normalized to the PMN-PT transducer, the commercial device is 40 dB lower in signal intensity. This is good evidence that the PC-MUT transducer should be able to penetrate much deeper into the SiC medium, and produce larger echoes based on flaws. It must also be noted that the commercial device was focused, and the composite transducer was not, implying that even more contrast could be gained for the TRS transducer. There are also no matching layers currently on the device.

The center frequency of both signals is lower than the expected 75 MHz, though this could be due to a number of factors, including the frequency dependent attenuation in water [13]. This accounts for approximately 6 dB of signal intensity. Electrical mismatch could also be a factor, as the impedance drops at higher frequencies, and more electrical transmission losses could occur. This is currently being studied. The bandwidth of both transducers is near 90%, and the -20 dB pulse lengths are 60 ns and 55 ns for the commercial and composite transducers, respectively. Pulse-echo experiments for 75 MHz PC-MUT were then conducted with water path decreased from ~ 5 nm to ~ 0.4 mm between the transducer front and the target surface (Figure 10, Figure 11). The center frequency is increased to 62 MHz comparing with ~ 45 MHz in Figure 10), the bandwidth is about 80%, and the sensitivity increasing is also noticed. The commercial transducer was not tested at shorter water path since it is a focused transducer and the response decreased drastically as the target is away from its focal point.



Figure 10. Time domain and spectral magnitude of echo signals for 75 MHz transducers. Comparison of a commercial device (focused) and TRS composite device show the composite gives much greater signal strength with comparable bandwidth.



Figure 11. Time domain and spectral magnitude of echo response from TRS 75 MHz transducer at distance of approximately 0.4 mm from the SiC echo target.

3.2 C-scan Experiments

3.2.1 C-Scan experiments using a 10 MHz composite transducer

In order to compare the relative performance of these transducers, a 13 mm thick sintered SiC tile was used for c-scan experiments. Measurements were taken every 200 μ m in order to form a high resolution image of the sample ultrasound response. At each point the bottom surface longitudinal signal amplitude, the longitudinal wave's time of flight, and the bottom surface shear signal amplitude were recorded. For the longitudinal measurements, a receiver gain of 10 dB was used for the TRS 10 MHz transducer while a gain of 15 dB was used for a commercial 10 MHz transducer (1/4" aperture, flat).

The C-scan images of longitudinal amplitude measured by each transducer can be seen in Figure 12. In these images, the TRS transducer appears much more responsive to variations within the sample, easily resolving a few anomalous points as well as regions of high and low amplitude. Due to its increased sensitivity and finer resolution, the C-scan image produced using the TRS 10 MHz transducer is clearly superior and will provide better data for quantitative analysis. The longitudinal time of flight image can be seen in Figure 13. The TRS transducer's C-scan image shows a clear variation within the sample without the inclusion of non-representative patterns.



Figure 12. Bottom surface longitudinal reflected signal amplitude for both transducers on a tile of sintered SiC. Scales are in mV.





3.2.2 C-Scan experiments using 75 MHz PC-MUT

In order to illustrate the performance of the TRS 75 MHz transducer, C-scans were performed measuring the longitudinal amplitude and time of flight for a few different samples including a 13 mm thick sintered SiC tile and a 4 mm thick disc of CVD SiC disc. These same tiles were characterized using a commercial 75 MHz transducer (non-focused) in order to form a performance comparison.

The comparative images for amplitude and time of flight for the first 13 mm thick SiC tile can be seen in Figures 14 and 15, respectively. In both of these Figures, the TRS 75 MHz transducer shows remarkably more sensitivity to individual features while exhibiting finer resolution than the images obtained using the commercial 75 MHz transducer. Some noise points are seen in the time of flight image for the TRS transducer (Figure 15), typically around areas which, in Figure 14, show anomalous features or low amplitude. This is due to the signal being lost at some points. The loss of a few points will not significantly impact either a qualitative evaluation or a quantitative analysis. The C-scan images of the 4mm thick CVD SiC disc can be seen in Figure 16. For this sample, the TRS 75 MHz transducer is able to locate small variations which are not visible using the commercial 75 MHz transducer. The TRS 75 MHz transducer's images show finer detail allowing for a better understanding of the material.



Figure 14. Bottom surface longitudinal reflected signal amplitude for both transducers on a tile of sintered SiC. Scales are in mV.



Figure 15. Bottom surface longitudinal time of flight for both transducers on a tile of sintered SiC. Scales are in µs.



Figure 16. Bottom surface longitudinal reflected signal amplitude and time of flight for both transducers on a disc of CVD SiC. Scales are in mV for amplitude and μs for TOF.

4. CONCLUSIONS

PMN-PT single crystal/epoxy 1-3 composites with frequency of 10 MHz, 15 MHz, and 75 MHz were successfully fabricated using the dice-and-fill and PC-MUT techniques. PMN-PT single crystal composites showed electromechanical coupling coefficients of ~0.67-0.79, and the loss remains low (< 0.06). The coupling coefficient of the piezo materials used in the commercially high frequency transducers is < 0.5. The prototyped composite transducers exhibited high sensitivity and broad bandwidth, which was confirmed by the C-Scan imaging experiments. The TRS transducers demonstrated the capability of NDE imaging with high resolutions in the detection of small inhomogeneities within a CVD SiC sample as bulk ceramic samples.

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