## Single Crystals for High Frequency Ultrasound

Kevin A. Snook<sup>1</sup>, Xiaoning Jiang<sup>1</sup>, Wesley S. Hackenberger<sup>1</sup>, Xuecang Geng<sup>2</sup>

<sup>1</sup>TRS Technologies, State College, PA 16801 <sup>2</sup>Blatek, Inc., State College, PA 16801

### ABSTRACT

Bandwidth is of integral importance in ultrasonic imaging due to its relationship to spatial resolution and is of even more significance in high frequency (HF) ultrasound, as it enables techniques to broaden its applicability. In medical ultrasound both harmonic imaging and improved depth of penetration have been enabled by wide bandwidth transducers. Composite piezoelectric ceramics provide a significant improvement in bandwidth over conventional bulk materials, due to the higher electromechanical coupling from the decrease in clamping and the better acoustic matching of the composite structure. Commercial composites have been historically limited to 10 MHz, due to the constraints of dicing technology and grain-pullout in ceramics. Single crystal PMN-PT provides a step forward in this technology due to its significantly higher coupling  $(k_{33} > 90\%)$ .

This paper gives an overview of ongoing research at TRS Technologies regarding development of single crystal PMN-PT 1-3 composites. Composites up to 15 MHz have been made using a traditional dice-and-fill technique and composites in the range of 20-50 MHz have been developed using a novel processing approach. Composites made from this method have effective electromechanical coupling near 75%. Results from these composites will be reported.

### A. INTRODUCTION

Ultrasound imaging has become instrumental in medical diagnostics, both in conventional applications such as fetal and gastrointestinal imaging and in newer areas such as skin imaging (dermatology). To overcome the inherent limitations of ultrasound, namely frequency dependent attenuation and resolution, current research is focused on increasing the frequency content of transducers (bandwidth), efficiency and sensitivity. Increased bandwidth not only improves resolution, but also has allowed for implementation of techniques to increase depth of penetration [1] or image contrast, and has increased the relevance of ultrasound towards diagnosis of skin tumors such as cutaneous melanoma. Imaging methods such as sub-harmonic or harmonic imaging have also led to better detection of Doppler flow and improved visualization of the vasculature.

The active piezoelectric is the most important factor in achieving good performance, and a number of groups have evaluated ferroelectric polymers, ceramics and single crystals as medical imaging transducers [1-3]. Conventionally, transducers with wider bandwidths incorporate piezoelectrics with larger mechanical losses and lower electromechanical coupling, such as poly(vinylidene fluoride), that must be driven at high levels to achieve a desirable signal-to-noise ratio.

Both 2-2 and 1-3 piezoelectric composites provide a viable alternative to high loss piezopolymers. The decreased clamping condition in the piezoelectric yields an improved electromechanical coupling coefficient, which inherently increases the bandwidth [4]. The composites also exhibit a better acoustic impedance match to tissue than bulk materials, which increases energy transmission into the tissue. The two main limitations of composites has typically been the scale and dimensioning requirements of the kerfs and posts, and the fact that composites using conventional ceramics can only improve the bandwidth by approximately 20%.

Single crystals such as PMN-PT or PZN-PT have inherently higher electromechanical coupling than ceramics, and provide a means to significantly increase transducer bandwidth over 100%. Novel methods of fabrication such as laser micromachining, tape casting and extrusion, and improved technology in conventional dicing have also enabled the means to achieve the scale necessary for operation above 10 MHz. By utilizing both

Method	PZT Fibers	Interdigital	Injection	Tape Casting	Stacking
Connectivity	1-3	2-2	1-3/2-2	2-2	2-2
Freq. (MHz)	50	40	< 10	30	30
Vol. %	45	90	> 40	84	75
k <sub>eff</sub>	0.70	0.66	NA	0.62	0.67
v <sub>1</sub> (m/s)	3800	4400	NA	4900	4050

Table 1. Comparison of high frequency piezoelectric composites made using different methods.

of these aspects, devices with improved imaging characteristics can be realized [5]. The specific aim of this work is the development of composites utilizing single crystal PMN-PT for improved transducer performance. A review of composite fabrication techniques is given, and current work regarding 2-2 and 1-3 composites are discussed. A new method of composite fabrication has shown effective coupling near 80%.

# Review of Composite Technologies

Current commercial composites for low frequency ultrasound utilize 2-2 and 1-3 PZT ceramic composites manufactured with traditional dicing saw techniques. Though easy to fabricate, PZT ceramics are limited in the bandwidth they provide. This cost-effective technique is advantageous for low frequency medical imaging

A number of methods have been developed to produce piezoelectric composites that utilize active polymer, ceramic and crystal materials. A few groups have looked at diced single crystal materials at lower frequencies, though there is little data on transducers made from these since chipping is an issue. Meyer *et al* [6] developed a 1-3 composite using drawn PZT rods made from a sol gel process. They developed 50 MHz transducers with up to 70% coupling, though the strain varied across the aperture due to the non-uniform spacing and relatively low (< 50%) volume fraction of PZT posts. Laser micromachining and injection molding shows promise for 1-3 and 2-2 composites, though this work is in its early stage [7-9].

Techniques for development of 2-2 composites include tape casting, stacking and interdigital bonding. The interdigital bonding technique achieved a high volume fraction, though proper alignment is crucial and may be difficult at higher frequencies [10]. Ritter *et al* made 30

MHz stacked composites with a coupling of 67% [4], though the process is extremely time intensive and requires good thickness control. A few groups have also developed tape casting techniques for composites [11, 12], which show promise for high frequency composites. A comparison of the various techniques is shown in Table 1.

These techniques may improve transducer performance, however, a number of techniques cannot be readily adapted to single crystal materials. It is also difficult to determine the maximum frequency these materials can attain. Since single crystals have the ability to achieve better performance, improving or developing techniques to create composites using the single crystal can establish a significant advantage.

# **B. PMN-PT 1-3 COMPOSITES**

Composite plates were formed using two different approaches, the first being a more conventional dice-and-fill approach and the second being a proprietary processing technique for higher frequency production.

# Conventional Composite Design and Results

Dicing of single crystals is much more difficult than ceramics, since the lack of domain boundaries permits cracks to propagate through the material freely. Since traditional dicing essentially cracks and chips away at materials, cracks along certain orientations can quickly spread due to the stresses from the saw. Vibrations (or chatter) within the spindle can also create post breakage as the saw passes through the cut material.

Dicing was performed using 10-18  $\mu$ m wide blades from Disco Corp., and the entire 1-3 composite was diced at one time, to mimic a more production level process. The spindle speed and



Figure 1. Picture of PMN-PT posts directly after dicing.

Table 2. Diced composite properties as measured with direct measurements and impedance analysis.

Property	Value
Frequency	10 MHz
Vol. %	~56
$\epsilon^{T}/\epsilon_{0}$	1696
$\epsilon^{s}/\epsilon_{0}$	437
$\tan \partial$	0.014
k <sub>eff</sub>	0.80
$v_1 (m/s)$	3590

feed rate were varied to evaluate the conditions for the least macroscopic damage (i.e. chipping and cracking). Feed rates of 1 mm/sec were adequate to eliminate chipping, and ideal spindle speed varied with the thickness of blade. Thinner blades required speeds down to 3 krpm from above 20 krpm for thicker blades. This was because the combination of reduced bond material and higher speeds resulted in more diamond grit pullout as the blade exited the crystal, resulting in chipping on the backside of the blade.

A representative 10 MHz diced structure is shown in Figure 1. The dicing depth was greater than 250  $\mu$ m, and the maximum kerf width was approximately 22  $\mu$ m for the widest blade. The design is adequate for 10 MHz, though lateral modes between 20-25 MHz limit the performance above 15 MHz. Epo-Tek 301 epoxy (Epoxy Tech., Billerica, MA) was used as the inactive filler material. The epoxy was back-filled onto the composite structure and degassed, before curing overnight at room temperature to minimize expansion during curing.



Figure 2. The two-way pulse echo time-domain and spectral responses of the 10 MHz PMN-PT composite transducer.

Both sides of the composite were then lapped, and Cr-Au electrodes (500Å/1500Å) were applied to the faces. The disk was then diced into small plates and poled at 5 kV/cm. The measured properties are shown in Table 2.

A simple planar transducer was fabricated using a diameter of 2 mm. The device incorporated a centrifuged silver epoxy backing (E-SOLDER 3022, Von Roll Isola) which had an acoustic impedance near 6 Mrayls. The pulse echo response, shown in Figure 2, shows an extremely wide bandwidth (> 100%) and a fairly Gaussian timedomain envelope. Ringing is evident in the signal, which is most likely due to the lateral resonances since the 20 MHz peak in the spectrum corresponds well to the expected value.

### Novel Composite Design and Results

Since traditional dicing cannot achieve the feature scale necessary for operation above 20 MHz, and the other techniques are relatively time limiting and more appropriate for ceramics, a new technique more apt to single crystals was developed. Using the process, a nominal post width of 15  $\mu$ m was made while maintaining an aspect ratio (height/width) of three, with kerfs of 4-5  $\mu$ m around the posts. This kept the designed lateral modes within the kerf at

PMN-PT crystal (Cr/Au)



PMN-PT crystal after etching (Au electrode)



Figure 3. XRD of PMN-PT surface before and after processing, showing no change in the material.

Table 3. 40 MHz composite properties made using direct measurements and impedance analysis.

Property	Value
Frequency	40 MHz
Vol. %	~58
$\epsilon^{T}/\epsilon_{0}$	1800
$\epsilon^{\rm S}/\epsilon_{0}$	450
$\tan \partial$	0.02-0.03
k <sub>eff</sub>	0.73
$v_{l}(m/s)$	3000

twice the thickness mode resonance frequency.

The effects of the processing on the crystal were not known, and therefore the post structure was evaluated to determine whether there was any crystal degradation. Surface x-ray diffraction (XRD) showed no difference before and after the post fabrication, as shown in Figure 3. Strain measurements after processing also showed a  $d_{33}$  of over 1800 pC/N, which is very close to the bulk value. These results show that the process does not negatively affect the crystal structure, or alter the crystallographic orientation. It also does not introduce micro-cracking or chipping, which is still possible with most of the other methods.

Subsequent preparation of the samples was done using the same methods as outlined above



Figure 4. The two-way pulse echo time-domain and spectral responses of the 40 MHz PMN-PT composite transducer.

for the 10 MHz device. Small plates were diced out of the 30  $\mu$ m composite disk, and each was individually poled at 15 kV/cm at room temperature. Piezoelectric and elastic properties were measured or calculated using impedance analysis or direct strain measurements. A summary of the properties is shown in Table 3.

An unfocused, single element transducer with an active area of 1 mm<sup>2</sup> was fabricated using the composite. A conductive epoxy backing (E-SOLDER 3022) was employed, and no matching layer was used. The impedance was near  $100\Omega$  at the parallel resonance frequency.

Figure 4 depicts the two-way pulse echo time and spectral response of the PMN-PT 1-3 composite transducer reflecting off of a steel target. The 43 MHz center frequency agreed well with predictions, though the 65% bandwidth was slightly lower than desired. Compared to similar bulk ceramic devices with no matching layer, the bandwidth was better. This was due to the better acoustic matching and higher coupling. The bandwidth could have been affected by the lapping procedure, since the mechanical stress introduced to such a thin sample could have led to some microcracking. The high loss and larger poling field is evidence for this, and other methods of sample preparation are under investigation.

### **C. CONCLUSION**

PMN-PT single crystal has been used in a 1-3 piezoelectric composite, and high frequency single crystal transducers have been fabricated for testing. A traditional dice-and-fill technique was used to develop a 10 MHz composite, which showed a bandwidth of over 100% despite a lack of any acoustic matching layers. A volume fraction of near 60% was achieved, and chipping effects were negligible. A new processing method has also been developed to fabricate crystal composites that cannot be made using traditional methods: a 40 MHz composite with 58% volume fraction was fabricated and tested. Though mechanical stresses due to lapping could be creating microscopic damage, an electromechanical coupling above 70% was achieved and a resultant transducer showed 65% bandwidth with no matching layers. Both of these composite designs show significant promise for wide bandwidth medical imaging arrays and single elements, including use in harmonic imaging.

#### **D. REFERENCES**

- 1. C. Passmann and H. Ermert, *IEEE Trans. Ultrason. Ferro. Freq. Contr.*, vol. 43(4), pp. 545-552, 1996.
- F.S. Foster, M.Y. Zhang, Y.Q. Zhou, G. Liu, J. Mehi, E. Cherin, K.A. Harasiewicz, B.G. Starkoski, L. Zan, D.A. Knapik and S.L. Adamson, *Ultrasound in Med. & Biol.*, v. 28(9), pp. 1165-1172, 2002.

- K.A. Snook, J-Z. Zhao, C.H.F. Alves, J.M. Cannata, W-H. Chen, R.J. Meyer Jr., T.A. Ritter, K.K. Shung, *IEEE Trans. on Ultrason. Ferro. Freq. Contr.*, v. 49(2), pp. 169:176, 2002.
- T.A. Ritter, X. Geng, K.K. Shung, P.D. Lopath, S.-E. Park and T.R. Shrout, *IEEE Trans. Ultrason. Ferro. Freq. Contr.*, vol. 47(4), pp. 792-800, 2000.
- P. Marin-Franch, S. Cochran, K. Kirk, J. Mat. Sci.: Mat. In Electron., v. 15, pp. 715-720, 2004.
- R.J. Meyer Jr., S. Yoshikawa and T.R. Shrout, *Mat. Res. Innovat.*, v. 3, pp. 324-331, 2000.
- S. Corbett, T. Clary, B. Beck, B. Ross, P. Jordan, T. Hughes and J. Ketterl, *Proc. 1999 IEEE Ultrason. Symp.*, v. 2, pp. 1213-1216, 1999.
- L.J. Brown, RL. Gentilman, H.T. Pham, D.F. Fiore and K.W. French, *Proc. 2002 IEEE Ultrason. Symp.*, v. 1, pp. 499-502, 1993.
- M. Lukacs, M. Sayer and S. Foster, *Proc. 1997 IEEE Ultrason. Symp.*, v. 2, pp. 1709-1712, 1997.
- R. Liu, D. Knapik, K.A. Harasiewicz, and F.S. Foster, *Proc. 1999 IEEE Ultrason. Symp.*, v. 2, pp. 973-976, 1999.
- W. Hackenberger, S. Kwon, P. Rehrig, K. Snook, S. Rhee and X. Geng, *Proc. 2002 IEEE Ultrason. Symp.*, v. 2, pp. 1253-1256, 2002.
- R.P. Schaeffer, V.F. Janas and A. Safari, *Proc.* 1996 IEEE Ultrason. Symp., v. 1, pp. 557-560, 1996.