

2-1-2007

# A wideband PVDF-on-silicon ultrasonic transducer array with microspheres embedded low melting temperature alloy backing

Hyun-Joong Kim  
*Purdue University*

Hanwoo Lee  
*University of Minnesota*

Babak Ziaie  
*Birck Nanotechnology Center, Purdue University, bziaie@purdue.edu*

Follow this and additional works at: <http://docs.lib.purdue.edu/nanodocs>

---

Kim, Hyun-Joong; Lee, Hanwoo; and Ziaie, Babak, "A wideband PVDF-on-silicon ultrasonic transducer array with microspheres embedded low melting temperature alloy backing" (2007). *Other Nanotechnology Publications*. Paper 57.  
<http://docs.lib.purdue.edu/nanodocs/57>

This document has been made available through Purdue e-Pubs, a service of the Purdue University Libraries. Please contact [epubs@purdue.edu](mailto:epubs@purdue.edu) for additional information.

# A wideband PVDF-on-silicon ultrasonic transducer array with microspheres embedded low melting temperature alloy backing

Hyun-Joong Kim · Hanwoo Lee · Babak Ziaie

Published online: 11 November 2006  
© Springer Science + Business Media, LLC 2007

**Abstract** A PVDF-based 8-element ultrasound transducer array (1 mm × 1 mm element size with an inter-element spacing of 1 mm) on a silicon carrier substrate is fabricated and characterized. To improve the performance of the transducer, new CMOS-compatible fabrication technologies are introduced. These include: (1) adhesive micro-contact printing on non-radiating areas, and (2) glass microspheres (7–20 μm in diameter) embedded low melting temperature alloy (LMA) for backside electrical connection. The first improvement removes the adverse effects of adhesive layer (e.g., lower sensitivity) between the PVDF and backside contact while the second one improves the pulse-echo signal quality by eliminating reflections at the backing/water interface. The fabricated array elements are tested in a water tank and their pulse-echo response are recorded. The central frequency of each element is 25 MHz with a 100% measured 6-dB bandwidth (60% 3-dB bandwidth).

**Keywords** PVDF · Adhesive micro-contact printing · Ultrasonic transducer · Low-melting temperature alloy · Glass microspheres

## 1 Introduction

High-frequency (>20 MHz) ultrasound is an attractive modality for imaging fine tissue structures with promis-

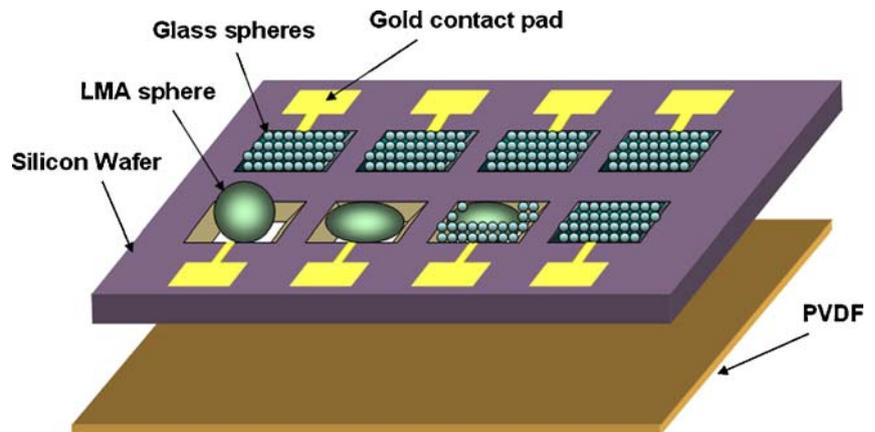
ing applications in ophthalmology, dermatology, and intravascular imaging (Passman and Ermet 1996; Ritter et al., 2002). Recent advances in transducer materials and micromachining technology have increased interest in high frequency transducer arrays for such applications. Several different transducer materials/technologies are being investigated for use in high-frequency ultrasound imaging. These include lead zirconate titanate (PZT), polyvinylidene fluoride (PVDF), and capacitive micromachined transducers (cMUT). PVDF, which is a piezoelectric polymer, offers several unique advantages compared to other materials. These include lower acoustic impedance, larger bandwidth, lower cost, and mechanical flexibility (Shung and Zippuro, 1996; Hunag et al., 2003). However, it has a lower sensitivity as compared to PZT. This can be overcome by integration of PVDF with active electronics. Micromachined PVDF arrays with active electronics on-board can be used in intra-vascular imaging with distinct advantages mentioned above. Several groups have reported on the fabrication of micromachined PVDF transducers, mostly attaching a PVDF film on the metallized (used for the bottom electrical contact) silicon substrate using epoxy and removing the silicon on the backside to improve sensitivity and reduce the cross talk (Swartz and Plummer, 1979; Mo et al., 1990). In order to successfully integrate PVDF with silicon substrate and active electronics, several major fabrication challenges need to be overcome. These include: (1) suitable backing material, (2) attachment methods to the substrate at low-enough temperatures (typically <70°C in order to retain the piezoelectric properties), and (3) avoiding organic solvents during the process (these damage the PVDF film). In addition, since PVDF film can not be easily patterned and etched, the array elements have to be defined in a suitable manner.

Among these, backing material selection is in particular critical, since this will determine the element center

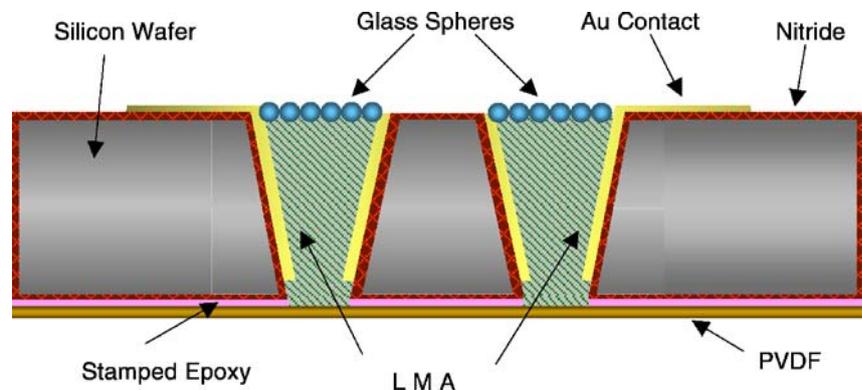
H.-J. Kim (✉) · B. Ziaie  
School of Electrical and Computer Engineering, Birck  
Nanotechnology Center, Purdue University,  
1205 W. State St., West Lafayette, IN 47907, USA

H. Lee  
Department of Electrical and Computer Engineering, University  
of Minnesota, 200 Union St SE, Minneapolis, MN 55455, USA

**Fig. 1** Schematic and components used in the fabrication of the transducer array



**Fig. 2** Cross section of the integrated transducer



frequency, bandwidth, and sensitivity. Each backing material has different acoustic properties such as acoustic impedance and attenuation. The optimum backing for wide-band applications should have matching acoustic impedance to that of the piezo-element while simultaneously having a large absorption coefficient. This is usually achieved at the expense of a lower sensitivity since a major part of the acoustic wave is lost and dissipated in the backing material. For PVDF transducers, the most common backing material has been conductive/silver epoxy (Fleischman et al., 2003). More recently, some investigators have experimented with tungsten powder filled epoxy in order to improve the backing performance (higher attenuation) (Grewe et al., 1990). Dispensing conductive epoxy can be done using automated dispensers or screen printing technique, however, both of these methods require dedicated expensive equipment and do not scale well to dimensions below  $100\ \mu\text{m} \times 100\ \mu\text{m}$  array element (5 nL backing volume assuming a  $500\ \mu\text{m}$  thick silicon wafer).

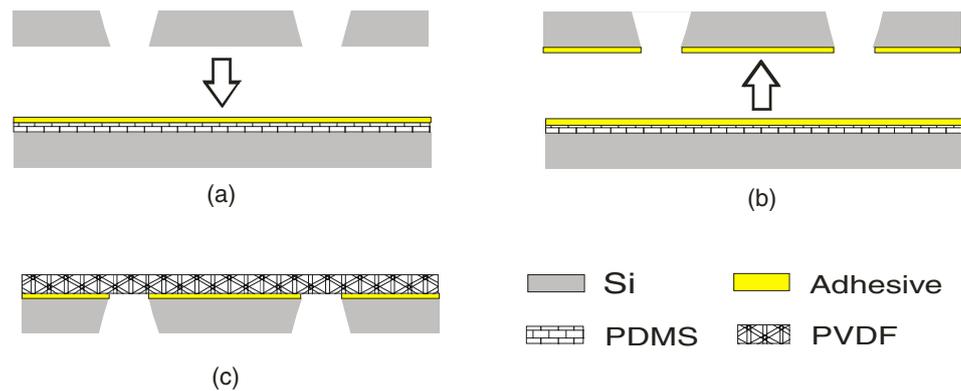
In this paper, we present new fabrication methods for high-frequency wideband ultrasonic transducer array that uses an epoxy stamped front surface for the attachment of a thin ( $9\ \mu\text{m}$ ) PVDF film and glass microsphere embedded low melting alloy (LMA) backing. The developed methods have several unique advantages including: (1) front-side patterning of adhesives using micro-contact printing,

(2) batch scale backside contact using LMA microspheres, (3) low processing temperatures, (4) increased scattering (reduced reflection) at the backing/water interface due to the irregular geometry using glass microspheres, (5) scalability through creating small LMA microspheres ( $\sim 100\text{--}300\ \mu\text{m}$  diameter), and (6) the potential for using self-assembly to fill the microspheres in small backing cavities (e.g.,  $100\ \mu\text{m} \times 100\ \mu\text{m} \times 100\ \mu\text{m}$ ).

## 2 Device structure and fabrication process

A general description of the array structure and fabrication process is given in this section. Figure 1 shows a schematic of the PVDF ultrasonic array structure and components used in the fabrication of the transducer. These include: (1) a silicon substrate with KOH-etched holes representing the array elements ( $1\ \text{mm} \times 1\ \text{mm}$  with inter spacing of  $1\ \text{mm}$ ), (2) a PVDF film coated with a thin gold layer on one side, (3) LMA spheres for backside electrical contact, and (4) glass microspheres for modifying the backing surface geometry. Figure 2 illustrates the cross section of the integrated ultrasonic transducer with a thin layer of glass microspheres covering the LMA backing surface. As will be discussed in Section 3, the presence of glass microspheres increases

**Fig. 3** Soft lithography stamping process (a) adhesive spin coated stamp (b) adhesive transferring on the Si substrate (c) detached PVDF film on the Si substrate

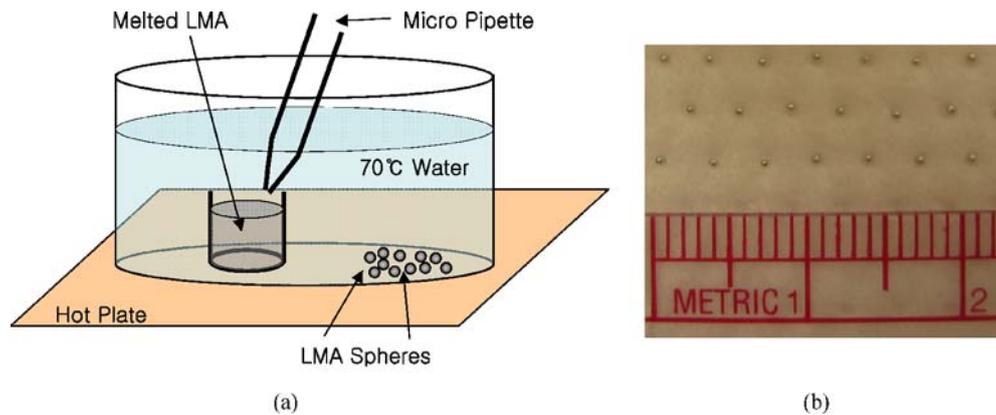


the scattering at the backing/water interface and reduces the back reflection at this surface, hence improving the pulse-echo response of the transducer.

The fabrication process starts with Low Pressure Chemical Vapor Deposition (LPCVD,  $\text{SiH}_2\text{Cl}_2:\text{NH}_3 = 100:20$  sccm at  $833^\circ\text{C}$  and 300 m Torr pressure, deposition rate  $\sim 40 \text{ \AA}/\text{min}$ , Thermco LPCVD) of low-stress silicon nitride ( $\text{Si}_3\text{N}_4$ ) on a *p*-type  $\langle 100 \rangle$  silicon wafer. The nitride layer ( $\sim 1 \mu\text{m}$  in thickness) is used as a mask during the KOH etching process. After a standard lithography process (Mask # 1), the silicon nitride on the backside of wafer is patterned ( $1.7 \text{ mm} \times 1.7 \text{ mm}$  openings) using a reactive ion etcher (STS etcher with a  $\text{CF}_4:\text{O}_2 = 400:4$  sccm plasma at 100 m Torr pressure and 100 W power level resulting in an etch rate  $\sim 700 \text{ \AA}/\text{min}$ ). After patterning silicon nitride, silicon wafer is etched throughout its thickness ( $500 \mu\text{m}$ ) in 45% KOH solution for 12 hr at  $80^\circ\text{C}$  (etch rate  $\sim 0.7 \mu\text{m}/\text{min}$ ) to generate  $1 \text{ mm} \times 1 \text{ mm}$  openings on the front side. Silicon nitride on the top and the backside is then stripped using a dry etcher (same recipe as the above). This process is done in order to remove any dielectric layer between the PVDF (which will be attached later) and the conductive backing material (which will be filled subsequently). A Plasma Enhanced Chemical Vapor Deposition (PECVD,  $\text{NH}_3:\text{N}_2:\text{SiH}_4:\text{He} = 2.5:520:140:140$  sccm plasma at  $340^\circ\text{C}$  and 900 m Torr pressure with 10 W power, Plasma-Therm 790) silicon nitride ( $3000 \text{ \AA}$  in thickness) insulation layer is then deposited to cover the front, back, and sidewalls of the KOH-etched silicon wafer. Next, a Ti/Au ( $300 \text{ \AA}/3000 \text{ \AA}$ ) layer is deposited (E-beam evaporator, CHA, deposition rate  $3 \text{ \AA}/\text{sec}$ ) on the nitride coated backside of the wafer and patterned through a standard lithographic process (Mask #2) and wet etching. This layer is necessary in order to provide a better connection to the measurement system subsequent to the backside filling. The PVDF film is next attached to the front side of the silicon substrate using stamped epoxy as an adhesive layer. In this step, a one-side Ti/Au ( $100 \text{ \AA}/1000 \text{ \AA}$ ) coated PVDF ( $9 \mu\text{m}$  in thickness corresponding to 125 MHz free space resonant frequency, Precision acoustics Ltd.) film

is prepared separately. Figure 3 shows a soft lithography stamping process to attach the PVDF film to the silicon substrate (Love et al., 2001). During this process, a polymeric stamp is first fabricated through spin coating (3000 rpm for 30 sec) and curing ( $100^\circ\text{C}$  oven for 20 min) a PDMS (Sylgard 184, Dow Corning) layer on a silicon wafer. Subsequently, the PDMS stamp is spin-coated with a thin adhesive layer (6000 rpm for 40 sec,  $< 10 \mu\text{m}$  thick, Eccobond 24, Emerson and Cumming Corp.) Fig. 3(a) Spin-coated adhesive is then transferred to the front side of silicon substrate through stamping process Fig. 3(b). The PVDF film (non-metallic side) is subsequently bonded to the adhesive transferred side of the substrate, Fig. 3(c). The thickness of the stamped adhesive can be controlled by changing the spin speed. This method allows for a clean and repeatable transfer of adhesive epoxy to the silicon substrate without clogging the KOH-etched pits. After attachment, the PVDF film is loaded into a vacuum chamber for 10 min and subsequently cured at room temperature for 24 h. Finally, the wafer is diced (DISCO wafer dicing saw) to separate the transducer  $8 \times 1$  arrays.

Low melting temperature ( $47^\circ\text{C}$ ) alloy spheres ( $\sim 100\text{--}300 \mu\text{m}$  diameter) are manufactured for backing and electrical connection to the ultrasonic transducer. These LMA spheres are fabricated through dispensing a specified amount of melted LMA (Bismuth 44.7%, Lead 22.6%, Tin 8.3%, Cadmium 5.3%, Indium 19.1%, Small parts Inc.) droplets into a hot water ( $70^\circ\text{C}$ ) bath (Fig. 4(a)). The dispensed alloy assumes a spherical shape throughout its dissensions in the water bath (similar to the old method for making cannonballs by dropping melted lead through shot towers). Due to its high surface tension and small size, the spheres keep their shape even after touching the bottom of the water bath. The hot water is then cooled down to the room temperature to solidify the spheres. Figure 4(b) shows a photograph of manufactured LMA spheres which have a diameter of approximately  $100\text{--}300 \mu\text{m}$ . The backside electrical contact to the PVDF is achieved via filling the KOH-etched holes with these LMA spheres and melting them on a hotplate. In the melted state,

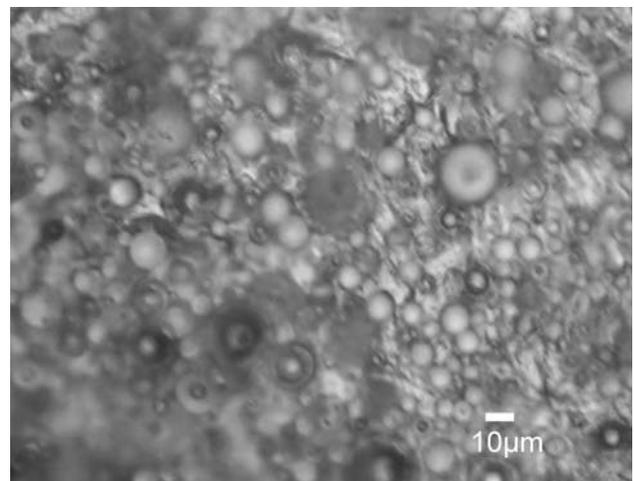


**Fig. 4** (a) Experimental set up for manufacturing low-melting temperature alloy spheres (b) Manufactured low-melting temperature alloy microspheres ( $\sim 100$ – $300\mu\text{m}$  in diameter)

glass microspheres ( $7$ – $20\ \mu\text{m}$  in diameter, Sigma-Aldrich) are dispersed onto the surface of the melted alloy backing in order to modify/roughen the surface geometry, hence increasing the signal scattering at the backing/water interface. This is followed by cooling the device to the room temperature, hence solidifying the alloy and glass microspheres in place. Figure 5 shows the surface of the glass microspheres embedded LMA backing (it is difficult to focus on the LMA surface due to its irregular and non-planar nature). As can be seen, dispersed glass microspheres self-spread on the surface of the melted LMA backing. Due to the low density of glass microspheres, dispersed spheres float on the melted LMA surface and generate a mono layer. Figure 6 is a photograph of fabricated 8-element ultrasonic transducer array. Each element has a gold trace pad that is used to wire bond the array to the 8-channel diplexer board for measuring the pulse-echo signal.

### 3 Test results and discussion

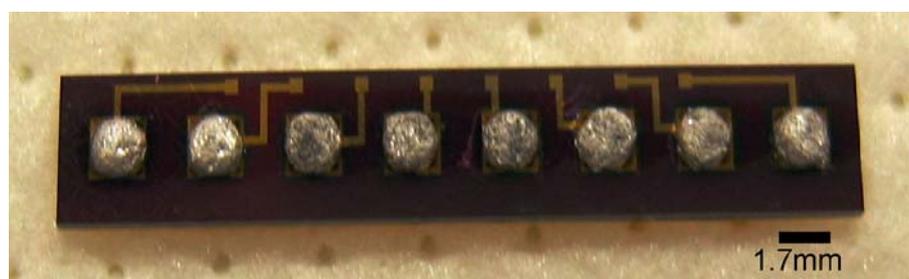
Figure 7 illustrates the experimental setup for measuring the pulse-echo signal. The measurement of the array elements was performed using a GE panametrics 5900 HF pulser-receiver. A thick (2 cm) flat Plexiglas plate was immersed at various depths inside a large water tank. An 8-channel diplexer was used to select one of the 8-elements and a



**Fig. 5** A photograph of the LMA backing surface with embedded glass microspheres

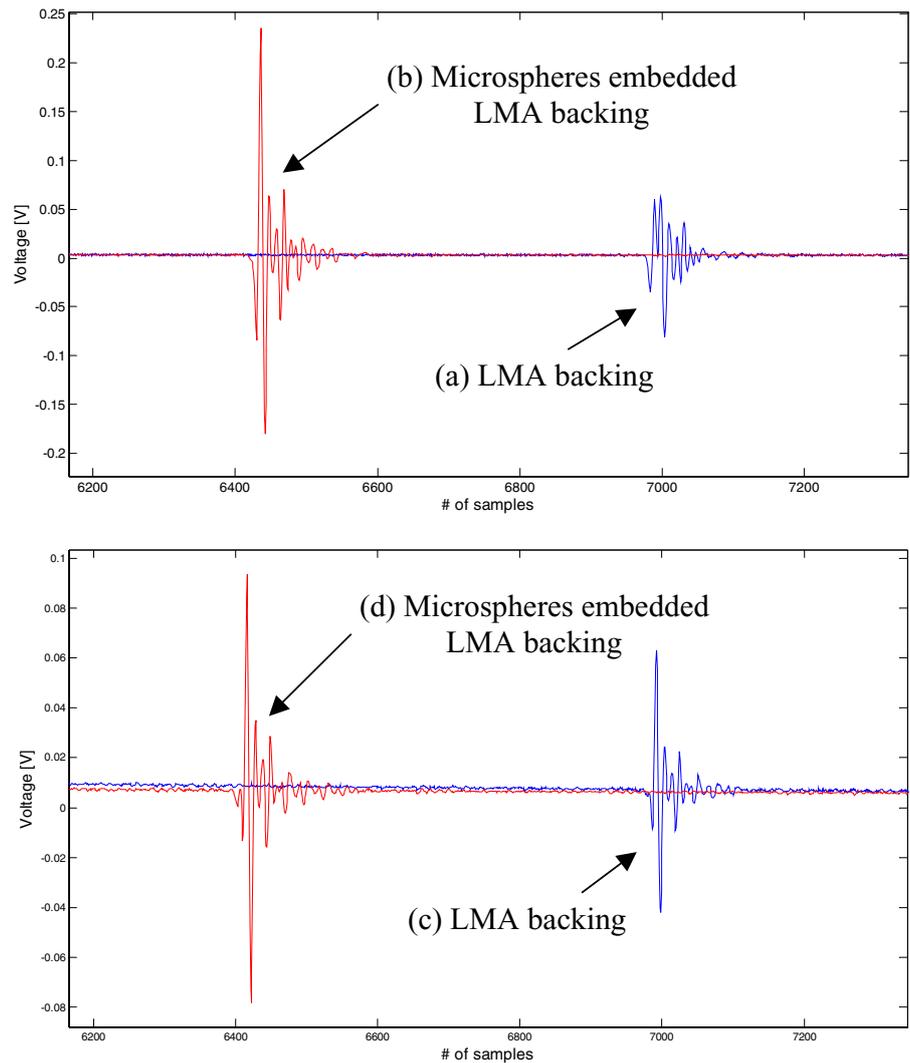
1 GHz Tektronix Gage digitizer was used to sample the receiver output. Figure 8 shows a pulse-echo signal of one of 8-elements in the time domain. The array element was excited by a broadband signal (up to 200 MHz) with  $1\ \mu\text{J}$  (minimum) to  $32\ \mu\text{J}$  (maximum) energy (100–200 V peak signal). Pulse repetition rate was set to 1 KHz. Figure 8(a) and (c) show the response of transducer array elements without glass microspheres (only low melting temperature alloy backing), whereas (b, d) show the pulse-echo response of

**Fig. 6** Photograph of an 8-element transducer array





**Fig. 8** Measured pulse-echo signals with plain LMA backing (a, c) and with glass microspheres embedded LMA backing (b, d)



coefficient ( $R$ ) due to the acoustic mismatch can be calculated using:

$$R = \left| \frac{Z_0 - Z_L}{Z_0 + Z_L} \right| = 0.7 \quad (2)$$

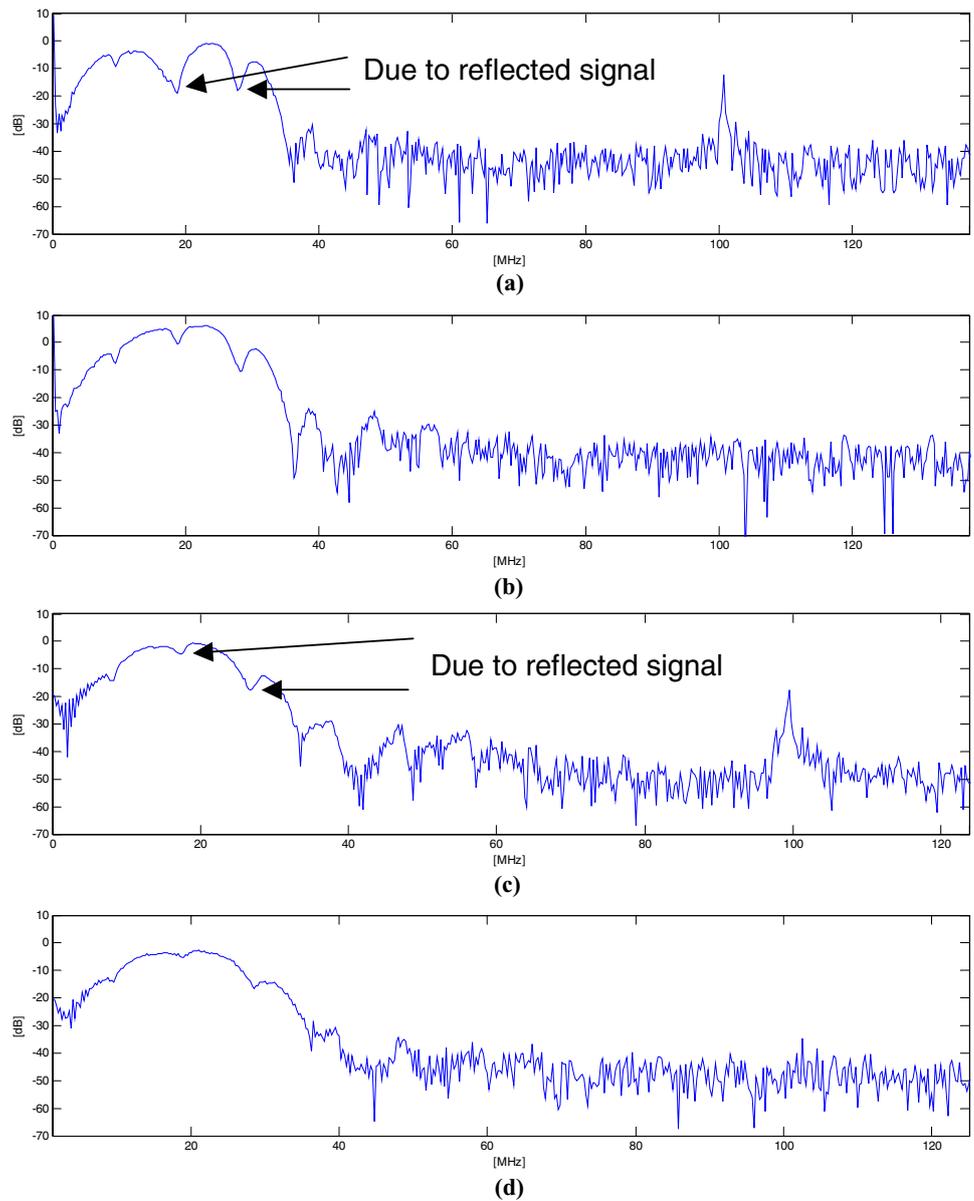
where  $Z_0$  is the acoustic impedance of PVDF ( $\sim 4 \text{ MRayl}$ ) and  $Z_L$  is that of the LMA. Acoustic impedance of tungsten-filled-epoxy is between 6 to 36 MRayl depending on the loading ratio (Shung and Zippuro, 1996). As can be seen, the LMA can have an unfavorable impedance matching property compared to that of conductive epoxies. However, the BW and sensitivity of the PVDF transducers reported in the literature using conductive epoxies are comparable to that of our LMA backing (Sleva et al., 1996). This indicates that the metal loading ratio commonly used in order to enhance the electrical properties tend to increase the acoustic impedance and brings it closer to that of the LMAs.

Another reflection happens at the LMA/water interface. Due to the limited thickness of the LMA backing ( $500 \mu\text{m}$ ) compared to the larger conductive epoxy elements, certain fraction of wave is reflected from the LMA/water interface. The reflection coefficient at this interface is  $\sim 0.88$  (acoustic impedance of water is  $\sim 1.54 \text{ MRayl}$ ). As can be seen, almost the entire incident signal is reflected from the boundary between the LMA and water resulting in the destructive interference signal loss at harmonics corresponding to integer multiples of the wavelength in the LMA (see Figs. 9(a) and (c)). These dips in the spectrum occur at wavelengths-thickness corresponding to:

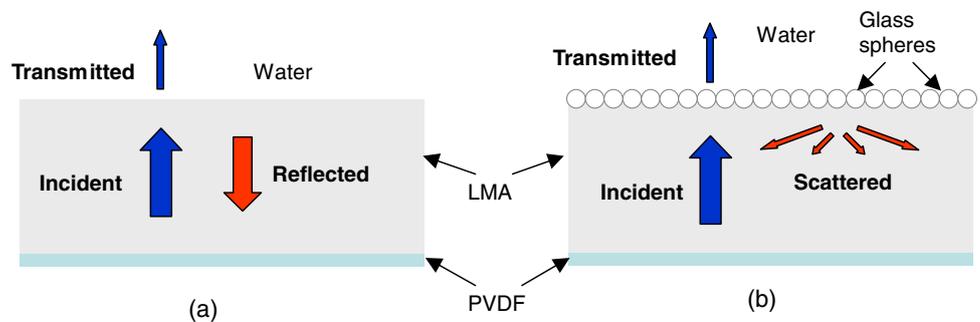
$$T = 2n\lambda, \quad n = 1, 2, 3. \quad (3)$$

where  $T$  is thickness of backing ( $\sim 500 \mu\text{m}$ ) and  $\lambda (= c/f)$  is wavelength. The calculated destructive interference frequencies (9, 18 and 27 MHz) corresponds accurately to the

**Fig. 9** Frequency domain echo response of plain LMA backing (a, c) and with glass microspheres embedded LMA backing (b, d)



**Fig. 10** Schematic of the acoustic signal propagation (reflection, transmission, and scattering) (a) plain LMA backing, and (b) glass microspheres embedded LMA backing



measured frequency dips in the spectrum. This power loss can have a detrimental effect on the transducer performance resulting in its lower sensitivity as is noticed in Fig. 8. Irregular LMA/water interface geometry, due to the dispersed glass

microspheres, enhances signal scattering and reduces direct back reflection at this interface. Figure 10 illustrates signal propagation and reflection in (a) plain LMA backing and (b) glass microspheres embedded LMA backing. The echo

signal of glass microspheres embedded LMA backing transducer shows greater amplitude than the plain LMA backing one (Fig. 7). In addition, as can be seen in Fig. 8(b) and (d), the harmonic dips in the spectrum are reduced in the glass microspheres embedded LMA backing transducer.

#### 4 Conclusion

In this paper, a PVDF-on-Silicon ultrasonic transducer array was fabricated and characterized. Our major goal was to introduce new CMOS-compatible and scalable fabrication technologies to manufacture integrated PVDF wideband transducers for high frequency imaging applications. These technologies included: (1) adhesive micro contact printing, and (2) glass microspheres embedded low melting temperature alloy (LMA) material for backside electrical connection. The pulse-echo signal was measured with plain and glass microspheres embedded backing. Although no attempt was made to optimize the transducer performance, the reflected signal interference at backing was obviously reduced by adding glass microspheres. The transducer with glass microspheres embedded LMA backing having irregular surface geometry showed larger amplitude (higher sensitivity) in the time domain and less power loss at interfering harmonic frequencies. Although our array elements were rather large, we successfully fabricated  $100\ \mu\text{m}$  LMA microspheres which can be used to fill  $100 \times 100\ \mu\text{m}^2$  array elements using template-assisted self-assembly methods (Yin et al., 2001).

Using micropipettes with smaller dispensing capability would allow even smaller microspheres to be fabricated.

**Acknowledgments** The authors would like to thank Professor Emad S. Ebbini of University of Minnesota and Professor Eric S. Furgason of Purdue University for their valuable advice. We are grateful to staff of the Microtechnology Laboratory of the University of Minnesota and Purdue University.

#### References

- A. Fleischman, R. Modi, A. Nair, J. Talman, G. Lockwood, and S. Roy, *Sensors Actuators A* **103**, 76 (2003).
- M.G. Grewe, T.R. Gururaja, T.R. Shrojit, and R.E. Newnham, *IEEE Trans. Ultrason. Ferroelect. Freq. Contr.* **37**, 506 (1990).
- Y. Hunag, A.S. Ergun, E. Haeggstrom, M.H. Badi, and B.T. Khuri-Yakub, *IEEE J. MEMS* **12**, 128 (2003).
- J.C. Love, J.R. Anderson, and G.M. Whitesides, *MRS Bulletin* **26**, 523 (2001).
- J.H. Mo, J.B. Fowlkes, A.L. Robinson, and P.L. Carson, *IEEE Proc. Ultrason. Symposium* **1**, 323 (1990).
- C. Passman and H. Ermet, *IEEE Trans. Ultrason. Ferroelect. Freq. Contr.* **43**, 545 (1996).
- T.A. Ritter, T.R. Shrout, R. Tutwiler, and K.K. Shung, *IEEE Trans. Ultrason. Ferroelect. Freq. Contr.* **49**, 217 (2002).
- K.K. Shung and M. Zippuro, *IEEE Eng. Med. Biol. Magazine* **15**, 20 (1996).
- M.Z. Sleva, R.D. Briggs, and W.D. Hunt, *IEEE Trans. Ultrason. Ferroelect. Freq. Contr.* **43**, 257 (1996).
- R.G. Swartz and J.D. Plummer, *IEEE Tran. Electron Devices* **26**, 1921 (1979).
- Y. Yin, Y. Lu, B. Gates, and Y. Xia, *J. Am. Chem. Soc.* **123**, 8718 (2001). [http://www.panametrics-ndt.com/ndt/ndt\\_transducers](http://www.panametrics-ndt.com/ndt/ndt_transducers) (Technical notes, accessed in February 2006)