Ultrasound Transducers for High Pressure Environments Up to 1000 Bar

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Ultrasound Transducers for High Pressure Environments Up to 1000 Bar

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***Abstract*— This paper studies the influence of high ambient pressure on ultrasound transducers where the piezoelectric layer is bonded to the acoustic matching layer by two different methods: Au-Sn solid-liquid interdiffusion and standard polymer adhesives. The transducers were fabricated by bonding 6 MHz piezoelectric elements to silicon matching layers. Electrical impedance was measured on the transducers during exposure to 1000 bar pressure at 15 °C in a silicone oil filled pressure vessel. The bondlines of the transducers were characterized before and after pressure exposure by scanning acoustic microscopy and optical microscopy. The exposure to high pressure was found to have noticeable effects on the piezoelectric material of the transducers, whereas no degradation was observed in the bondlines.**

Keywords—Ultrasound, Transducers, Bonding, High Pressure.

I. INTRODUCTION

The development of ultrasound transducers for operation in high pressure, high temperature (HPHT) environments is still a challenge, mainly due to thermal aging effects of the piezoelectric material and the temperature limitations of polymer materials. The American Petroleum Institute defines HPHT environments as temperatures and pressures greater than 177 °C and 1030 bar [1].

Polymer composites are commonly used as acoustic matching and backing layers in ultrasound transducers and as bonding material in transducer stacks. The characteristic acoustic impedance of polymer-based materials is generally lower than that of piezoelectric ceramics. Large acoustic mismatch between the piezoelectric material and acoustic layers in the transducer stack leads to reverberations inside the transducer and limits its broadband characteristics [2].

Most organic materials cannot withstand temperatures above 300°C. In addition, differences in the coefficient of thermal expansion (CTE) of polymer composites and piezoelectric ceramics may cause deterioration of the ultrasound transducer after thermal cycling [3].

Solid-liquid interdiffusion (SLID) bonding [4], also called transient liquid phase bonding (TLP) [5], is a technique for creating hard metallurgical bonds. The technique involves two metals of high- and low-melting temperature (T_{high} and T_{low}) processed above T_{low}. Melting of the T_{low}-metal allows a relatively fast diffusion process, with chemical reactions yielding intermetallic compounds (IMCs). Such IMCs will be stable at temperatures above the processing temperature. The SLID bonding technique offers a well-defined, metallurgical bondline with excellent mechanical strength and electrical conductivity, which is well suited for die attach and interconnections in high temperature electronics [6].

The present work is motivated through the assembly of ultrasound transducers for HPHT applications by the Au-Sn SLID technique. The high melting temperature of the IMCs makes it suitable for operation in high temperature environments and the characteristic acoustic impedance of the metallurgical bondline is acoustically beneficial compared to softer polymeric bondlines. Furthermore, Tollefsen et. al. [7] proved that Au-Sn bonds can absorb CTE induced thermo-mechanical stresses during thermal cycling. This stress absorption is beneficial as crack formations in the piezoelectric may deteriorate the performance of the transducer. One challenge related to SLID bonding for ultrasound transducers is the formation of voids in IMCs. Our previous work [8] reported insignificant acoustic effects of voids occupying up to 20% of the Au-Sn bondline for ultrasound transducers up to 10 MHz, whereas 15% delamination of the width of the bondline would introduce noticeable effects for frequencies above 5 MHz. In HPHT environments, voids may have considerable impact on the mechanical strength of the bond, hence the performance of the ultrasound transducer. IMCs are normally brittle [9] and the existence of voids in these layers may lead to crack development in the bondline under high ambient pressure.

This paper compares transducers bonded with standard epoxy and with the Au-Sn SLID technique. A piezoelectric material was bonded to silicon dies, which were chosen for this work to act as the first matching layer in a multi-matching layer stack. Transducers were characterized by electrical impedance during exposure to 1000 bar pressure to investigate effects of high ambient pressure on the bondlines.

II. Method

A. Sample Preparation

Piezoelectric elements (L-155N, Tayca Corp.) of 10 mm × 10 mm were bonded to Au-plated Si substrates of 20 mm × 20 mm. Two-component epoxy (3M Scotch-Weld Epoxy Adhesive DP460, 3M Company) was used for the epoxy-bonded samples where a bonding jig was used to apply 2 ± 0.1 MPa bonding force during curing for 3 hours at 60 °C. Au-Sn SLID bonds were formed by placing an 25 µm thick 80 wt% Au – 20 wt% Sn eutectic preform between the piezoelectric material and a Si substrate electroplated with between 7 and 13 µm Au. This stack was placed in the bonding jig where spring pins applied 0.5 ± 0.1 MPa bonding force to the stack during bonding. Fig. 1 illustrates the bonding jigs used for the two bonding techniques. The full fabrication process for the SLID bonded samples is detailed in related work [10].

The piezoelectric elements of the SLID bonded transducers were depoled during the SLID bonding process, due to the high process temperature of 310 °C. The SLID transducers were repoled by DC-stepping up to 460 VDC in room temperature for 1 minute. Repoling of the epoxy bonded samples was not necessary, as the bonding temperature did not reach the Curie temperature of the piezoelectric material.

The structures were diced into two equal halves in order to evaluate the bonding layer. Finally, Cu wires of length 10 cm were connected to the electrodes on the PZT layer using silver epoxy.

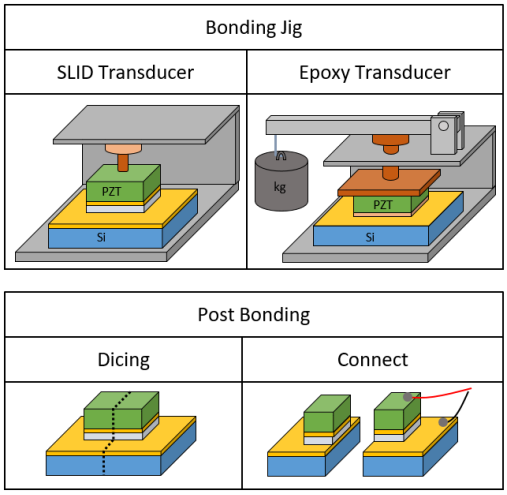

Fig. 1 – Bonding Process and Sample Preparation Steps.

B. Experimental Setup

The transducers were put into a pressure vessel filled with silicone oil, and a manual pressure pump was used to regulate the pressure inside the vessel. The transducers were electrically connected to a network analyzer through a sealed connector in the pressure vessel. Fig. 2 illustrates the experimental setup. The network analyzer was calibrated without this connector, but the inductance introduced by the connector and the Cu wires did not influence the measurements at the frequencies investigated.

Three testing schemes were used for exposing the transducers to 1000 bar ambient pressure at 15 °C, as listed in Table 1. After exposure to pressure, the samples were cleaned in an ultrasonic bath to remove residue oil.

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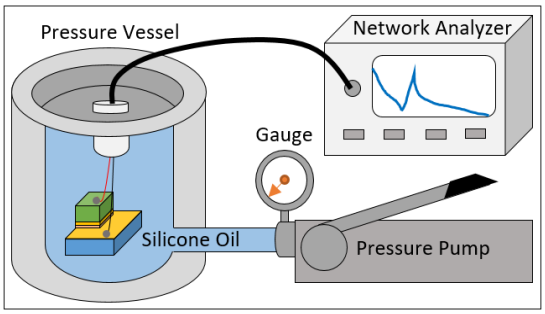

Fig. 2 - Pressure testing setup with silicone oil filled vessel, pump, gauge and network analyzer.

TABLE 1 – TESTING SCHEMES

Testing Scheme		Transducers Tested	
		SLID	***Epoxy***
I	One 5-minute exposure at 1000 bar.	2	2
II	Three 15-minute exposures at 1000 bar.	1	1
III	13 hours at 1000 bar.	1	1

C. Sample Characterization

The transducers were investigated by acoustical and optical microscopy before and after exposure to 1000 bar ambient pressure. A scanning acoustic microscope (SAM 300, PVA TePla) with a 150 MHz focused ultrasound transducer was used to make C-scans with an 8 ns window at various depths in the sample. Cross-sections of the transducer stack were prepared by Ar ion-milling (Hitachi, IM4000), and the cross-sections were inspected using an optical microscope (Neophot 32, Carl Zeiss Jena). Electrical impedance was measured in air using network analyzers before and after exposure to high pressure (ZVL13, Rhode Schwarz) as well as during pressure testing at various pressures (HP 8751A, Keysight Technologies).

III. Results

Twelve transducers were prepared, six using epoxy and six using SLID bonding. Layers and thicknesses are listed in Table 2. Five transducers of each kind were exposed to 1000 bar pressure while one was kept for reference. In addition, two PZT samples with dimensions 10 mm × 10 mm were exposed to pressure.

The cross-sectional micrographs in Fig. 3 show the bondlines of the two transducer types after exposure to high pressure. The maximum thickness of the epoxy bondline is 3 µm, with sections of direct contact between the PZT and the Si matching layer. The total SLID bondline thickness is 27 ± 3 µm. Voids of up to 5 µm diameter are observed in the bondline.

TABLE 2 – TRANSDUCER LAYERS

Layers		Thicknesses [µm]	
SLID	***Epoxy***	***SLID***	***Epoxy***
PZT		320	
Cr Sputtered		0.02	
Au Sputtered		0.2	
Au Electroplated	Epoxy	10	3
AuSn Preform		25	
Au Electroplated		10	
Au Sputtered		0.2	
Cr Sputtered		0.02	
Si		540	

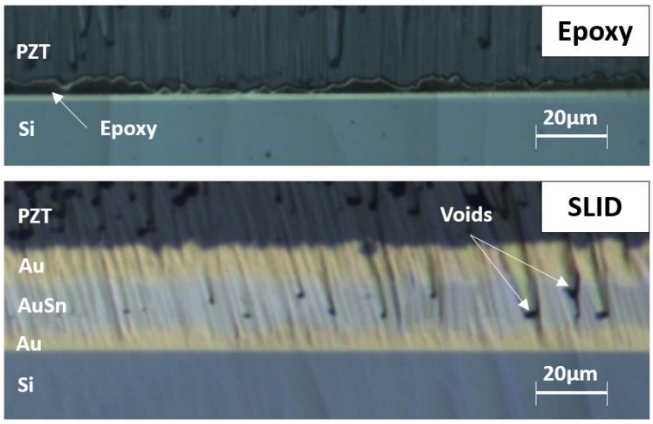

Fig. 3 - Cross sectional micrograph of the epoxy transducer (top) and the SLID transducer (bottom) after exposure. The SLID bondline contains voids which did not develop crack formations due to pressure exposure.

The electrical impedance of the PZT samples without matching layers, seen in Fig. 4, shows a small change in impedance magnitude with pressure. This change is almost reversible, settling close to the original impedance magnitude when the pressure is released. A 16 % reduction in impedance magnitude at anti-resonance frequency is the most evident change after pressure exposure (Fig 4, right plot).

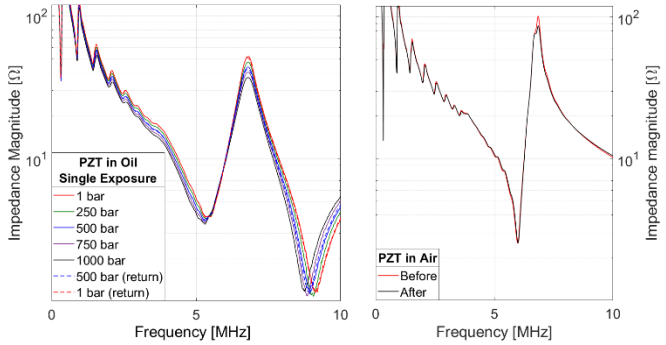

Fig. 4 – Piezoelectric element exposed to one single exposure of 1000 bar. Left figure shows electrical impedance curves of a PZT sample for increasing pressures up to 1000 bar. Right figure shows measurements in air before and after exposure to pressure.

One transducer of each kind was exposed to 1000 bar for 13 hours. Results are shown in Fig. 5. Only very small changes were visible after returning to 1 bar after 13 hours pressure exposure, but a small reduction in the impedance magnitude can be observed, most pronounced around the anti-resonance frequency (Fig. 5, right plots).

One transducer of each kind was exposed to three repeated exposures to 1000 bar. Only very small changes were seen from the repeated exposures. A degradation was most notable for the SLID transducer, whereas the epoxy transducer show a slightly reduced impedance magnitude around anti-resonance.

Inspection in the scanning acoustic microscope (SAM) revealed cracks formed in the PZT of the SLID transducers, as seen in the middle image in Fig. 7. The left image shows a top view photography of the SAM-inspected sample, while the left image shows the SAM and photographic images overlaid each other. Such crack formations were visible in several of the SLID bonded transducers and were caused by the spring-pin of the jig used during bonding.

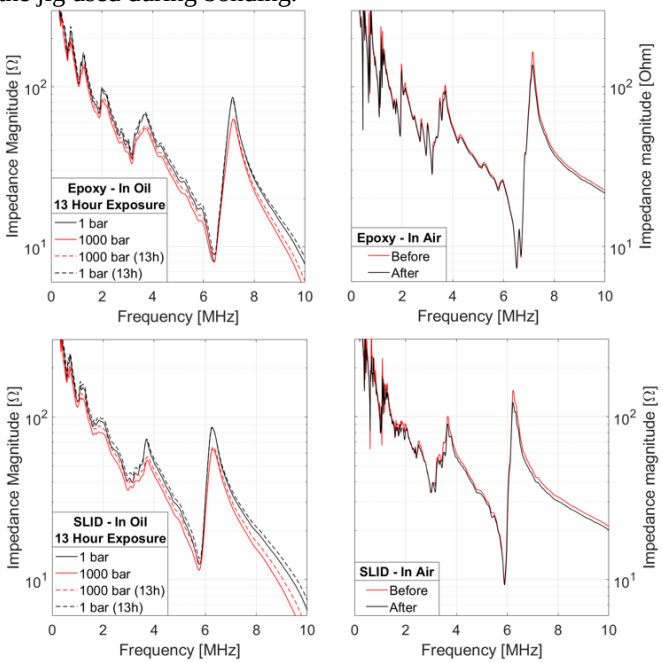

Fig. 5 – Epoxy (top figures) and SLID (bottom figures) were exposed to 1000 bar for 13 hours. Left figures show the electrical impedance measured during pressure exposure in silicone oil. Right figures shows before and after measurements in air.

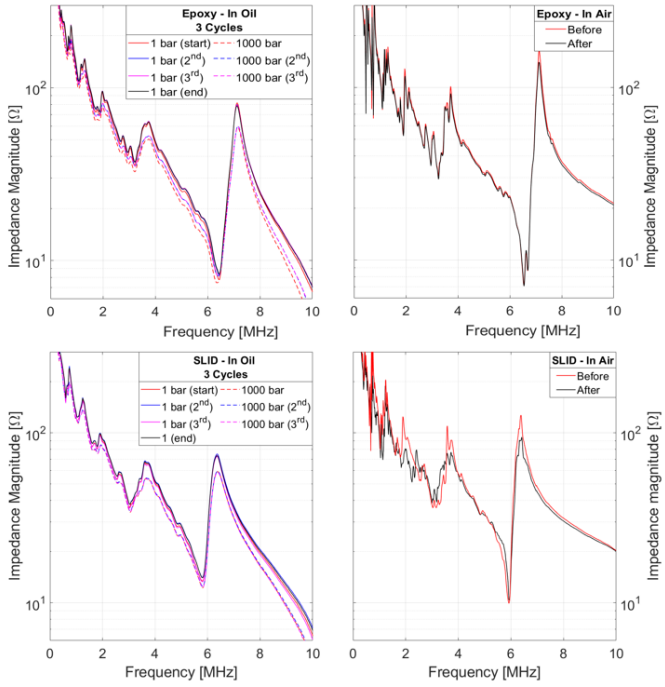

Fig. 6 – One epoxy transducer (top figures) and one SLID transducer (bottom figures) were exposed to three repeated exposures to 1000 bar. The left figures show the measurements during pressure testing in silicone oil. Right figures show electrical impedance in air before and after exposure.

Figure 7 – Crack formations in a SLID transducer. Top-view photography of the sample (left image), SAM-image of the same sample (middle image), and the SAM-image overlaid the photography (right image).

IV. Discussion

The bondline in the epoxy bonded transducers is thin, approximately 3 µm, with point contact between electrodes on the Si substrate and the piezoelectric material. The SLID bondline thickness was approximately 27 µm and contained small voids. As shown in our previous work [8], intermetallic Au-Sn bondlines of these dimensions has negligible influence on the electro-acoustic transfer function of the transducer due to the increased characteristic acoustic impedance. Our measurements found no change in the voids contained in the intermetallic bondline after exposure to pressure.

The Curie temperature of the PZT material used in this work is 150°C, while the bonding process used for the Au-Sn SLID system reaches 310°C. This made it necessary to repole the PZT after assembling the SLID transducers. The epoxy transducers were cured at temperature 60°C, and no repoling was needed for these transducers. From Fig. 5 and 6 it is seen that the epoxy transducers were slightly more resonant than the SLID transducers, suggesting a higher Q-factor. This degradation of the Q-factor of the SLID transducers may be explained by imperfect repoling of the piezoelectric ceramic in these transducers. Hence, piezoelectric materials with higher Curie temperatures is of interest for further work.

All measurements found that the electrical impedance magnitude shifted down during exposure to pressure. Under compressive stress, the piezoelectric material partially depoles as the domain alignment is reoriented. This is known to degrade the Q-factor and cause an increase of the dielectric permittivity [11]. Releasing the pressure decreased the permittivity of the piezoelectric material back to original levels, with a 16 % permanent reduction of the electrical impedance magnitude at anti-resonance. This reduction of magnitude was caused by a decrease in the Q-factor of the piezoelectric material.

Repeated exposures to high pressure had more impact on the SLID transducer than the epoxy bonded transducer. The degradation of this SLID transducer is believed to be caused by the effects of repoling, as no visible changes were observed in the bondline with microscopy techniques.

The crack formation in one of the SLID transducers, shown in Fig. 7, was caused by direct contact between the spring of the bonding jig and the piezoelectric element. The high localized bonding pressure on the brittle ceramic caused multiple small cracks to form during transducer manufacturing. This is a consequence of the high bonding pressure needed to manufacture the SLID bonds, and may be eliminated by using a structural layer to distribute the force more evenly, as done with the epoxy bonded transducers.

V. Conclusion

We have studied the effects of high pressure exposure on ultrasound transducers made with Au-Sn SLID bonding and epoxy bonding. Transducers were exposed to 1000 bar pressure and inspected for degradation. Electrical impedance was sensitive to pressure-induced changes in the piezoelectric material and was in combination with scanning acoustic microscopy found to be an efficient characterization technique. All transducers successfully passed the 1000 bar ambient pressure test, although slight reductions in the Q-factor at anti-resonance were observed. No changes were observed in the bondline of the transducers, suggesting that both bonding techniques are suitable for operation at high pressures.

Acknowledgment

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