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Impact of High Pressures on Au-Sn Solid Liquid Interdiffusion (SLID) Bonds

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***Abstract* — This paper investigates the influence of high pressure on Au-Sn solid-liquid interdiffusion (SLID) bonds formed by bonding Si substrates to dies of either lead-zirconate titanate (PZT) with high surface roughness or Si with low surface roughness. Bonded samples were exposed to 1000 bar pressure in a silicone oil filled pressure vessel. Samples were characterized before and after exposure by means of scanning acoustic microscopy, optical microscopy and scanning electron microscopy with energy dispersive x-ray spectroscopy. All but one sample successfully passed the pressure exposure. This failed sample had a delamination in the proximity of a large void in the intermetallic layer.**

Keywords—SLID, Intermetallics, High Pressure, Ultrasound, Piezoelectric

I. INTRODUCTION

Solid-liquid interdiffusion (SLID) bonding [1], also called transient liquid phase bonding (TLP) [2], 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 interconnection technology for high temperature electronics [3].

Previous work at University of South-Eastern Norway has proven the feasibility of Au–Sn SLID bonding of Si-based and SiC samples, where >78 MPa die shear strength was achieved [4]. More recently, the Au-Sn SLID technique was proven feasible for bonding acoustic backing layers to piezoelectric material in ultrasound transducers using PZT-ceramic [5].

Lead-Zirconate Titanate (PZT) is commonly used as the piezoelectric material in sensor applications such as pressure sensing or ultrasound transducers for well inspection in the oil and gas industry where temperatures and pressures are considerably high. American Petroleum Institute defines high pressure, high temperature environments as temperatures and pressures greater than 177 °C and 1030 bar, respectively [6]. The development of ultrasound transducers for operation in harsh environments is still a challenge, mainly due to thermal aging effects of the piezoelectric material and the temperature limitations of polymer materials. Polymer composites are commonly used to make acoustic matching and backing layers in ultrasound transducers and for bonding acoustic layers to the piezoelectric material. Most organic materials cannot withstand temperatures above 300 °C. In addition, difference in the coefficient of thermal expansion (CTE) of polymer composites and piezoelectric ceramic may cause deterioration of the ultrasound transducer after thermal cycling [7].

The present work is motivated through the assembly of ultrasound transducers for high-temperature and high-pressure applications by the Au-Sn SLID technique. The high melting temperature of the IMCs makes it suitable for operation at high temperature environments and the characteristic acoustic impedance of the metallurgical bondline is acoustically beneficial compared to softer polymeric bondlines. Furthermore, Tollefsen et. al. [4] proved that Au-Sn bonds absorb CTE induced thermo-mechanical stresses during thermal cycling.

One challenge related to SLID bonding for ultrasound transducers is the surface roughness of the mating surfaces, which may pose challenges with wetting and affect the formation of voids in IMCs. The surface roughness of the PZT is determined by the grain-size and is for most conventional PZT ceramics in the range of 3 to 5 μm [8]. In high frequency ultrasound applications, single crystal ferroelectric materials, such as Lead Magnesium Niobate-Lead Titanate (PMN-PT), are preferred where the surface roughness can be machined comparable to that of Si. Our previous work [9] 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 high-pressure environments, voids may have considerable impact on the mechanical strength of the bond, hence the performance of the ultrasound transducer. IMCs are normally brittle [10] and the existence of voids in these layers may lead to crack development in the bondline under pressure.

This paper compares the bonds formed by bonding Si substrates to dies of either PZT with high surface roughness or Si with low surface roughness. Si dies were chosen for this work to emulate a piezoelectric material with low surface roughness such as PMN-PT since such materials were not readily available for this study. Bonded samples were exposed to 1000 bar pressure to investigate effects of pressure on bondlines containing voids. Influence of temperature during pressure exposure is not considered in this paper.

II. METHOD

A. Sample Preparation

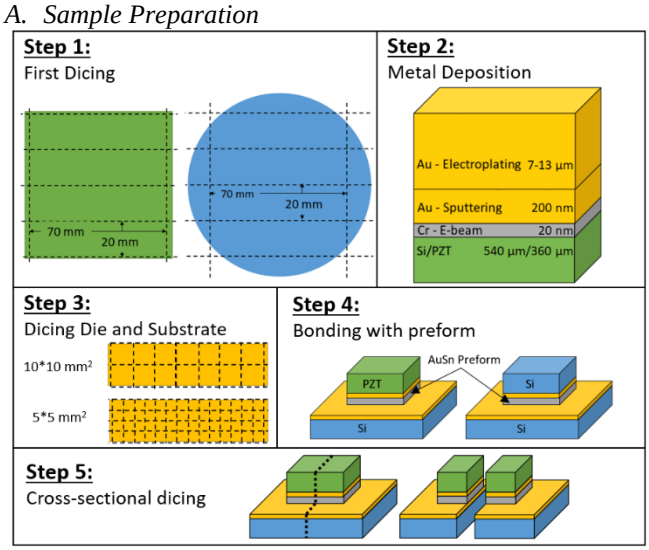

Fig. 1 – Sample preparation steps.

Two sample groups consisting of Si substrates and dies of either PZT or Si, were prepared according to Fig. 1. Si and PZT samples were plated by layering 20 nm Cr, 200 nm Au, then electroplating 7-13 µm Au. A profilometer (Vecco, Dektak 150) measured the absolute surface roughness, R_T, of the samples by line scans before and after Au electroplating. The plated samples of Si and PZT were diced into substrates of 10 x 10 mm^2 and dies of 5 x 5 mm^2.

Samples were bonded using 25 µm eutectic Au 80 wt% - Sn 20 wt% preform. The substrates and dies were fixed in a bonding jig, shown in Fig. 2, with a spring-pin mechanism to set the bonding force, as listed in Table 1. A vacuum solder system (Budatec, VS160 UG) was used for bonding. A thermal conductive sheet was placed between the bonding jig and the hotplate for improved heat conduction. A temperature sensor was attached to the base of the jig to record the bonding profile, shown in Fig. 3.

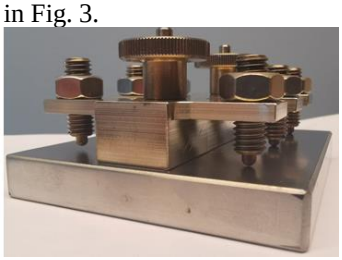

Fig. 2 - Bonding jig with spring loaded pins for setting the bonding pressure.

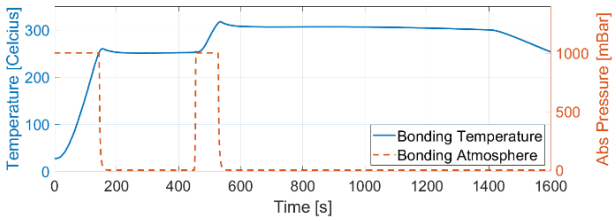

Fig. 3 – Bonding profile showing bonding temperature and bonding atmosphere.

TABLE 1 – SAMPLE DESCRIPTION AND BONDING PRESSURE

Substrate	Die	Bonding pressure	Number of Diced Samples
Si	Si	0.6 ± 0.1 MPa	6
Si	PZT	0.5 ± 0.1 MPa	6

Samples were first annealed for 5 minutes at 250 °C before a temperature increase to 310 °C for a 15 minutes bonding step. During the temperature ramping steps, the bonding atmosphere of nitrogen was raised to ambient pressure to achieve a faster temperature ramp rate of 105 °C/min, representative to that of our previous work [5]. The complete cooling step is not shown in Fig. 2 as this step was performed outside of the vacuum solder system by placing the bonding jig on a heat sink for rapid cooling.

After bonding, each sample was diced into two equal parts to allow for inspection of the cross-section before and after exposure to high pressure.

B. High Pressure Test Setup

The diced samples were exposed to 1000 bar pressure at 15 °C. The pressure vessel was filled with silicone oil and a manual pressure pump was used to regulate the pressure in the vessel, shown in Fig. 4. Three testing schemes were used, as listed in Table 3. After exposure, the samples were cleaned in an ultrasonic bath to remove residue oil.

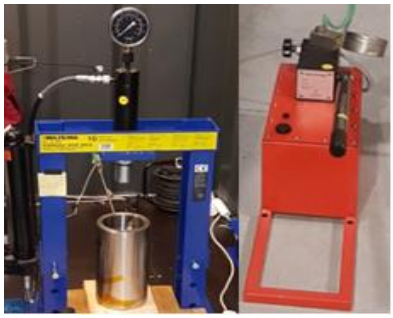

Fig. 3 – Setup for pressure testing. A manual pump with a pressure gauge pressurized the vessel filled with silicone oil.

TABLE 2 – HIGH PRESSURE TESTING SCHEMES

	Testing Scheme	Number of Diced Samples
I	Single exposure: 5 minutes at 1000 bar.	Si-Si: 2 PZT-Si: 2
II	Pressure cycling: Three exposures of 15 minutes at 1000 bar.	Si-Si: 2 PZT-Si: 2
III	Long exposure: 13 hours at 1000 bar.	Si-Si: 1 PZT-Si: 1

C. Characterization Techniques

Bonded samples were characterized by optical- and acoustic microscopy before and after exposure to high pressure. A scanning acoustic microscope (PVA TePla, SAM 300) was used to inspect each sample before dicing, step 5 of Fig. 1, and for inspecting samples before and after exposure to high pressure. A 150 MHz focused ultrasound transducer was used to make several C-scans with an 8 ns window at various depths in the sample. SAM C-scans are a non-destructive technique for examining inside the sample, where the window size determines the depth range of focus. Under these operating conditions, this SAM can detect voids wider than 30 µm and delamination between layers.

Cross-sections, showing bondlines of test samples, were prepared by means of Ar ion-milling (Hitachi, IM4000). The bondline inspection was carried out using an optical microscope (Carl Zeiss Jena, Neophot 32) and a SEM-EDX (Hitachi, SU3500). EDX analysis measured the metallurgical composition of the bondlines before and after exposure to high pressure.

III. Results

A. Au-Sn Bondline

The layered bondline consists of Au/ ζ' phase (Au_5Sn)/ Au layered structure. A complete conversion to the ζ' phase of the intermetallic layer was identified with no variation after exposure.

Fig.5 shows cross-section micrographs of the bonded samples of Si-Si (top) and Si-PZT (bottom) before exposure to pressure. The vertical lines observed in the micrographs were caused by the curtaining effect arising from the ion-milling process. The bondline thicknesses were 23 ± 2 µm for the Si-Si samples and 27 ± 2 µm for the PZT-Si samples. The measured surface roughnesses, listed in Table 3, indicated a significant increase in absolute surface roughness after electroplating the Au layer. This surface roughness can be observed through close inspection of Fig. 5. Small voids up to 5 µm diameter were observed scattered throughout the bondlines of every sample. These voids were located along the Au-IMC interfaces. Two of the Si-Si samples had large voids up to 50 µm wide in the center of the IMC, as seen in Fig. 6 and 7.

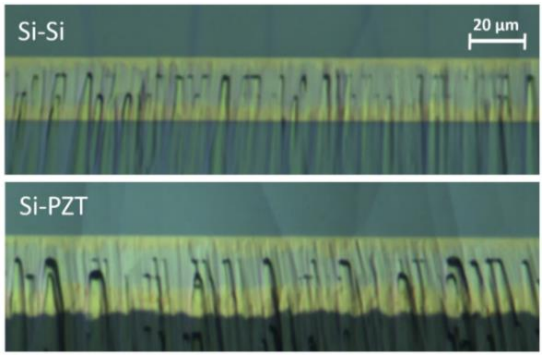

Fig. 4 – Cross-section micrographs of Si-Si sample (top) and PZT-Si sample (bottom) before exposure to pressure. The same magnification was used for both samples.

Table 3 - Measured Surface Roughness of Au

	Before Au plating		After Au plating	
	Si	***PZT***	***Au on Si***	***Au on PZT***
R_T [µm]	0.07	4.5	3.7	5.6

B. Si-Si Bonds

Five diced halves of Si-Si samples were exposed to high pressure while one was kept for reference. Fig. 6 shows cross-section micrographs of a Si-Si bonded sample before and after single exposure, testing scheme I.

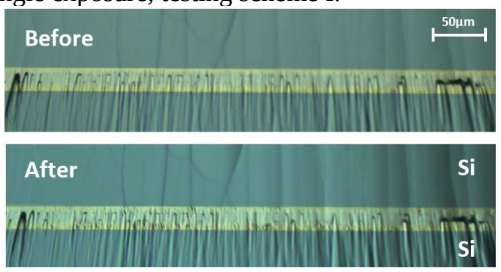

Fig. 5 - Cross-section micrographs of a Si-Si sample before and after a single pressure exposure, testing scheme I. The same magnification was used before and after.

Four of the Si-Si samples exposed to high pressure did not show any change after exposure. One Si-Si sample, shown in Fig. 7, had changed after exposure to high pressure cycling, testing scheme II. A delamination was visible at the interface between the Si substrate and die and the plated metallization layers. To the left of this large void, there was an enlarged void appearing after exposure. The delamination and the enlarged void was not visible on the SAM due to shadowing effects from the large void.

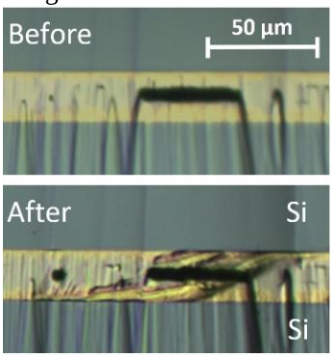

Fig. 6 - Cross-section micrographs of a Si-Si sample before and after pressure cycling testing scheme II.

C. PZT-Si Bonds

Cracking of the PZT dies caused by the fabrication process was evident from SAM scans in all the PZT-Si samples. The left and middle image of Fig. 8 show a PZT-Si sample imaged at different depths into the sample. The micrograph showed a large crack through the PZT. However, the bondline was not affected by the crack in the PZT.

Five diced halves of PZT-Si samples were exposed to high pressure while one was kept for reference. Fig. 9 shows a cross-section micrograph of a PZT-Si bonded sample before and after testing scheme II. No visible change was observed in any of the samples except for a darkening of the Ni electrode on the PZT, which had been deposited by the supplier. Fig. 10 shows SAM images before and after exposure to high pressure where no effects due to pressure exposure was observed. Signal intensity defines the signal amplitude received by the SAM, where a difference in signal intensity correlates with different material interfaces in the sample.

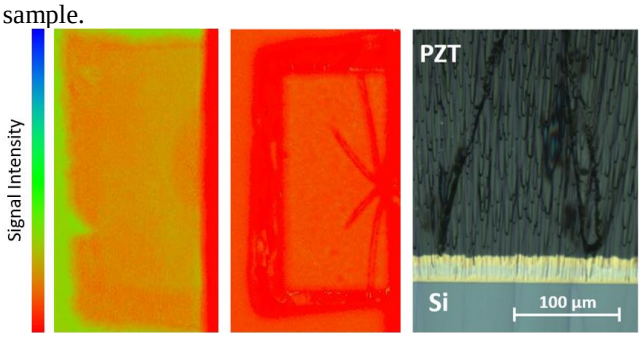

Fig. 7 - SAM images showing cracks in the PZT (left and middle) and a cross-section micrograph showing the crack through the PZT (right).

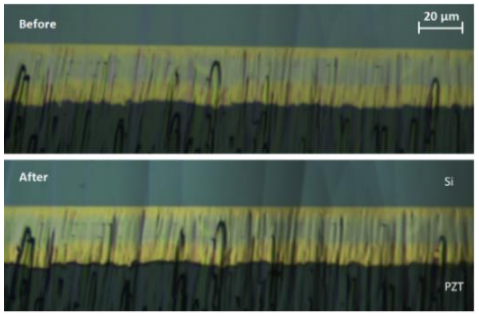

Fig. 8 - Cross-section micrographs of a PZT-Si sample before and after pressure cycling, testing scheme II. The same magnification was used before and after.

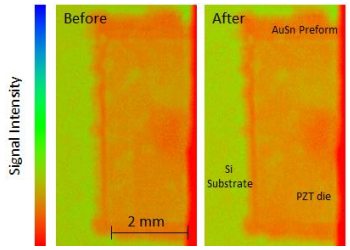

Fig. 9 – SAM images of a PZT-Si sample before and after exposure to high pressure

IV. Discussion

A. Au-Sn Bondline

Electroplating Au introduced considerable surface roughness on the Si and PZT surfaces, where the absolute surface roughness of the PZT was more pronounced than that of the Si. The voids in the IMC of Fig. 5 form defect planes and were larger for the PZT-Si samples. This may be explained by the higher surface roughness, which reduces wetting of the mating surfaces. Voids may also be caused by a volumetric change caused by the formation of Au_5Sn or due to the different diffusion rates of Au and Sn, known as the Kirkendall effect [11]. Nguyen et. al. [5] reported that a faster heating rate of 120°C/min reduced formations of long voids, which were typical for a slower heating rates of 20°C/min and 45°C/min. Fine-tuning the bonding process with a faster heating rate, longer bonding time and lower bonding temperature may reduce the formation of large 50 µm wide voids, as observed in the Si-Si bondlines of Fig. 6 and Fig. 7.

Based on previous studies [9], the acoustic performance of an ultrasound transducer would not be significantly impacted for low void concentrations of 5 µm in diameter. The void size and concentration found in the PZT-Si samples are not of critical concern for the acoustic performance of ultrasound applications up to 10 MHz. The large 50 µm voids found in the Si-Si samples would introduce noticeable effects for frequencies in the 5-10 MHz range.

B. Si-Si Samples

One Si-Si sample, shown in Fig. 7, had changed after exposure to high pressure. In the proximity of a large void located in an exposed section of the bondline, a delamination was visible at the interface between the Si substrate and die and the plated layers of Cr and Au. This suggests poor adhesion, but it is not clear which interface delaminated. The bondline was only partially delaminated and the bond seemed durable when attempting to apply shear force. To the left of the large void, one smaller void appeared enlarged after exposure. It is believed that this enlargement is caused by a deformation of a larger void inside the sample, this was however not confirmed. This sample was exposed to repeated cycles of pressure, suggesting that repeated exposures to high pressure is more critical to the bond integrity than single quick and single long exposures. The repeated stress from the high pressure cycling caused the voids in the bondline to deform. This increased stress around voids seemed to be relieved through delamination.

C. PZT-Si Samples

From the SAM characterization, it was evident that cracks had formed in the PZT during bonding. This crack formation was likely caused by the bonding jig, Fig. 2. The springs of the bonding jig push the samples together while bonding. These springs were in direct contact with the brittle PZT, presumably causing the PZT to crack from the localized stress. All samples of PZT dies had this characteristic. The SLID bondline was not affected by the crack and the bond was still intact after exposure to high pressure. Such cracks are undesired for ultrasound applications. This should be avoided in future experiments, e.g. by using a hard structural layer to distribute the force from the jig over the PZT die.

V. Conclusion

We have successfully demonstrated the feasibility of using the Au-Sn SLID technique for bonding surfaces of varying surface roughness, where the resulting bonds are suitable for operation up to 1000 bar pressure. All but one sample were unchanged after exposure to pressure. The sample failing the pressure test showed delamination close to a large void. This demonstrates the importance of good adhesion of the metallization layers on the samples. We conclude that the Au-Sn SLID technique is suitable for bonding ultrasound transducers for high-pressure applications.

Acknowledgment

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