HERMETIC INTEGRATION OF LIQUIDS IN MEMS BY ROOM TEMPERATURE, HIGH-SPEED PLUGGING OF LIQUID-FILLED CAVITIES AT WAFER LEVEL

M. Antelius, A.C. Fischer, F. Niklaus, G. Stemme and N. Roxhed
KTH – Royal Institute of Technology, Stockholm, Sweden

ABSTRACT

This paper reports a novel room temperature hermetic liquid sealing process based on wire bonded “plugs” over the access ports of liquid-filled cavities. The method enables liquids to be integrated already at the fabrication stage. Test vehicles were manufactured and used to investigate the mechanical and hermetic properties of the seals. A helium leak rate of better than 10^{-10} mbarL/s was measured on the successfully sealed structures. The bond strength of the “plugs” were similar to standard wire bonds on flat surfaces.

INTRODUCTION

Liquid-integration already at the microfabrication stage is a new enabling feature for MEMS with applications to life-science devices, high-sensitivity sensors where the liquid state can enhance sensing capabilities, and MEMS lenses. Specific examples include MEMS-based drug delivery systems [1] which are prefilled with drugs or protein solutions, liquid-based electrochemical sensors using liquid electrolytes which enable sensitivity levels in the ppb-range [2] and miniaturized optical lenses [3].

The difficulties related to liquid integration in MEMS often relate to the diminished temperature budget in processing which occurs when the liquid is integrated. This can be as low as 37 °C for sensitive or living materials in for instance life-science applications. In traditional wafer-level integration schemes the liquid is hermetically sealed inside the cavity during the wafer bonding process [4, 5, 6]. This forces the cavity formation and sealing, i.e. wafer bonding, to use room temperature processes such as adhesive wafer bonding [7].

Our group has previously shown wafer level room temperature hermetic liquid sealing by gold ring embossing [4], where a smaller gold ring on one wafer is compressed towards, and partially embedded in, a larger gold ring on the other wafer. We have also shown cold welding of overlapping gold sealing rings with negatively-sloped sidewall angles [5]. Both these methods seal the liquid in the cavity during wafer bonding and require additional mechanical stabilisation afterwards. This was implemented using polymer underfills. These methods have the advantage that they can be more hermetic than pure adhesive wafer bonding since the seal is metallic instead of polymeric. A potential limitation is the fact that the liquid was pipetted into every cavity using a serial process. This was recently addressed using a method of cavity formation and cavity sealing with the wafers submerged in the liquid to be integrated [6]. This method is however unsuitable when compared to serial pipetting for some applications. These applications include integrating solutions

Figure 1: Sketch of a sealed cavity with two plugged holes. The top silicon part is drawn transparent and a corner of the device has been cut away for better illustrating the seal.

that may pollute or alter surfaces, for instance surface fouling proteins, or when there is a risk of contaminating the liquid from the “immersion bonding” process itself. The pipetting method also has the advantage of being able to integrate two liquids, a necessity for the previously mentioned lens [3].

CONCEPT

In this work a method to fill and hermetically seal cavities at wafer level using small liquid access ports is introduced. The ports are used for filling and are sealed after the cavities have been formed. This alleviates the temperature restrictions on the cavity formation process since the liquid integration can be made during the final steps in the fabrication process. This method allows for both vacuum filling of the entire submerged wafer [8] or individual filling of each cavity by pipetting.

The method also enables the use of ultrasonic bonding since the access ports can be sealed individually. The liquid filled cavities are sealed at room temperature utilizing standard wire bonding where a gold wire “bump” is placed on top of an access port metallized with gold. Wire bonding is an extremely mature back-end technology with very high throughput [9]. This is also expressed in the cost of the process, for very high volume applications it has been reported to be on the order of 10 USD / 100,000 wire bonds [10]. Additional mechanical stabilization of the seal is not needed.

We demonstrate this method by sealing the access port to 50 μ l ($11 \times 11 \times 0.4$ mm) large bulk micromachined cavities. A drawing of the manufactured test vehicle with two sealed access ports is shown in figure 1. The tested access port diameters were between 20 and 55 μ m. This was chosen to fit the 25 μ m diameter bond wire which could form free air balls of up to around 75 μ m in diameter. The access ports were

Figure 2: (a) 400 μm deep KOH etching of 550 μm thick oxidized silicon wafers. (b) Anodic bonding and access port Deep RIE (c) Gold metallization followed by vacuum filling of water with red dye. (d) Gold wire bonding, “plugging”, on top of the access ports to the cavity, detailed in figure 3

placed over the sloping KOH-etched sidewalls, where the silicon is thicker. There might otherwise be a risk of breaking the silicon membrane since the wire bond capillary applies a pressure when the wire bond is formed. We tested designs with only two access ports and with up to 80 ports spread out along the edge of the cavity.

FABRICATION

A liquid sealing demonstrator was fabricated according to the process scheme indicated in figure 2. 100 mm diameter and 550 μm thick silicon wafers were used for cavity substrates. A hard mask was defined on the back side of the wafer in a thermally grown silicon dioxide layer using a photo resist mask and dry etching in an CHF_3/CF_4 -based plasma. 400 μm deep cavities were formed by KOH etching, followed by a wet silicon dioxide removal in buffered HF. The cavities were vacuum sealed by anodic bonding to a Pyrex wafer. Circular holes were deep reactive ion etched into the top silicon side down into the cavities using a 7 μm thick photo resist mask. In order to be able to wire bond in the holes, the top side of the wafer stack and the upper portion of the access ports were metallized by sputter deposition of 100/500 nm of TiW/Au. The open cavities were evacuated and filled [8] at wafer scale

Figure 3: The gold wire bond sealing process. A gold ball is formed by an electrical discharge, the ball is bonded to the substrate by force and ultrasonic energy applied with the bond capillary, the wire is sheared off by a horizontal motion of the bond capillary, yielding a sealed device.

with red dyed water. The wafer was then dried by blowing with nitrogen and wiping with a lint free cloth.

The filled cavities were sealed by “plugging” the access ports by wire bonding a “bump” at the top of the holes. The ball bonding process is illustrated in figure 3. A free air ball is initially formed at the end of the gold wire by an electrical discharge to the gold wire. The formed ball is bonded to the substrate with the aid of force and ultrasound. The bond capillary is then moved perpendicular to the device surface while still in contact with the bonded ball, thus shearing of the gold wire before moving away from the substrate.

The “plugging” was performed using a fully automated ESEC 3100+ wire bonder (ESEC Ltd, Switzerland) operating with a chuck temperature of 40 $^{\circ}\text{C}$. The wire bonder uses the so-called “bump module” software option that enables bump structures in an automated fashion without any special adjustment of the hardware. The wire bonding throughput during processing was up to 15 plugs/s without optimizing anything for improved speed. This translates to a rate of 7.5 cavities/s or in our case the full 100 mm wafer in less than 4 seconds. The primary speed limitation is the movement range of the bond head in the wire bonder.

RESULTS

The result of the wafer level cavity filling is shown in figure 4. Two 20 μm diameter access ports were enough for completely vacuum filling a cavity. One of the shown cavities is not filled since the corresponding access ports were not completely etched into the cavity. This was caused by the ports being small (20 μm) and additionally placed at the center of the wafer, resulting in a lower etch rate during the DRIE process. The result of the “plugging” process is illustrated in the SEM picture shown as figure 5. For comparison this figure also shows an empty 37 μm diameter access port. The used ball size of 75 μm proved to be sufficient to reliably seal holes smaller than 42 μm . The bump would shear off with the wire during the wire bonding for larger diameter holes. We do not expect any issues in moderately scaling up

Figure 4: Cavities filled with red dye, seen from the glass side. The empty cavity was not filled since the access hole was insufficiently deep etched. The dimensions of the cavities are 11×11 mm.

the gold wire diameter, and consequently the ball size. This would enable sealing of larger access port diameters. Our current limitation in wire diameter is related to the configuration of the wire bonder used.

The mechanical adhesion of ball bonds placed on holes with different hole sizes were investigated using a shear tester (2400PC, Dage Ltd., UK) where a metal blade applied an increasing force, perpendicular to the surface, on the “bump” until the bond broke. Bonds placed on unpatterned substrates were also measured for comparison. The average of at least five measurements and their standard deviation are plotted for hole diameters from 20 to $42 \mu\text{m}$ in figure 6. The increase in shear strength for larger hole diameters indicates that a significant amount of gold was pushed inside the hole during the bonding process. This was also confirmed by SEM imaging of a cross section of a sealed access port, shown in figure 7.

Figure 5: SEM picture of two fluidic access ports, with and without a wire bonded plug. The hole diameter is $37 \mu\text{m}$.

Figure 6: The shear strength of wire bonded plugs for different hole dimensions. Bonds on an unstructured surface is shown at $x = 0 \mu\text{m}$ for reference. Error bars correspond to 1 sigma.

A small constriction is also visible at the top of the access port. As is seen, this did not negatively impact the filling of the access port.

In order to investigate the hermeticity of the seals, the absolute leak rate was measured by a helium mass spectrometer (Pfeiffer Vacuum GmbH, Germany) attached to the backside of sealed through silicon holes (without cavities or the glass wafer). When using this method, called the “through hole method” [11], the amount of helium leaking through the hole from the atmosphere is analyzed. The connection to the chip was made using rubber o-rings with vacuum grease. This method only gives the current leak rate, although in a wide measurement range. Several blank tests were initially made

Figure 7: Cross sectional view of a wire bonded plug in $30 \mu\text{m}$ diameter fluid access port. The chip was embedded in PMMA and polished until the access port was reached.

Table 1: Measured helium leak rates for different hole diameters. Holes with diameters of less than 40 μm appears to result in hermetic seals.

Hole diameter [μm]	Leak rate [10^{-10} mbarL/s]
no hole	~ 1
24	1.4
37	1.2
44	5
48	5
52	74

using polished unstructured silicon pieces. All the results are shown in table 1. The leak rate for bonds on holes with a diameter less than 40 μm was measured to be better than the noise level of the leak detector, which is 1×10^{-10} mbarL/s. This leak rate level is three orders of magnitude better than the requirements on hermetic packages of this size according to the MIL standard 883F Test Method 1014.11. This methods pass criteria is related to having a long diffusion time for water to enter a cavity. The same is applicable here, but for a material flow in the other direction. Hence, the seal is considered to be hermetic for liquid encapsulation applications. The larger holes had increasingly larger leak rates. This is consistent with the behaviour seen during wire bonding, the yield of bonding on the larger holes was much poorer.

Sealed and diced devices were additionally burst tested by heating them slowly on a hot plate. At around 130 $^{\circ}\text{C}$ the lids of the devices started bulging outwards noticeably. At around 140 $^{\circ}\text{C}$ the 1.9 mm wide anodic bond surrounding the cavity failed, additionally indicating a good mechanical attachment of the gold seals.

CONCLUSIONS

We have described and demonstrated a novel room temperature liquid integration process using a wire bonding-based “plugging” process. The manufactured seals have been shown to be fully hermetic for liquid packaging applications. The seals were also thoroughly tested mechanically and were shown to be strongly attached. This method enables liquids to

be easily and efficiently integrated and packaged into MEMS devices at very low costs per seal.

REFERENCES

- [1] J. Santini, M. Cima, R. A. Langer, “A Controlled-release Microchip”, *Nature*, vol. 397, pp. 335–338, 1999.
- [2] E. Bakker, M. Telting-Diaz, “Electrochemical Sensors”, *Anal. Chem.*, vol. 74, pp. 2781–2800, 2002.
- [3] S. Kuiper, B. H. W. Hendriks, “Variable-focus Liquid Lens for Miniature Cameras”, *Appl. Phys. Lett.*, vol. 85, 1128, 2004.
- [4] M. A. Lapisa, F. Niklaus, G. Stemme, “Room-temperature Wafer-level Hermetic Sealing for Liquid Reservoirs by Gold Ring Embossing”, in *Digest Tech. Papers Transducers’09 Conference*, Denver, June 21–25 2009, pp. 833–836.
- [5] A. Decharat, J. Yu, M. Boers, G. Stemme, F. Niklaus, “Room-temperature sealing of microcavities by cold metal welding”, *J. Microelectromech. Syst.* vol. 18, pp. 1318–1325, 2009.
- [6] Y. Okayama, K. Nakahara, X. Arouette, T. Ninomiya, Y. Matsumoto, Y. Orimo, A. Hotta, M. Omiya, N. Miki, “Characterization of a Bonding-in-liquid Technique for Liquid Encapsulation into MEMS Devices”, *J. Microelectromech. Microeng.* vol. 20, 095018, 2010.
- [7] F. Niklaus, G. Stemme, J.-Q. Lu, R.J. Gutmann, “Adhesive Wafer Bonding”, *J. Appl. Phys.*, vol. 99, 031101, 2006.
- [8] J. Monahan, A. A. Gewirth, R. G. Nuzzo, “A Method for Filling Complex Polymeric Microfluidic Devices and Arrays”, *Anal. Chem.*, vol. 73, pp. 3193–3197, 2001.
- [9] G. Harman, *Wire Bonding in Microelectronics*, 2nd ed., McGraw-Hill Professional, 1997.
- [10] *3D IC Report*, Yole Development, February 2007.
- [11] De Wolf, A. Jourdain, P. De Moor, H. A. C. Tilmans, L. Marchand, “Hermeticity Testing and Failure Analysis of MEMS Packages”, in *Proc. of 14th Int. Symp. on the Physical and Failure Analysis of Integrated Circuits*, Bangalore, July 11–13 2007, pp. 147–154.