SPHERICAL RUBIDIUM VAPOR CELLS FABRICATED BY MICRO GLASS BLOWING

E. Jesper Eklund[1], Andrei M. Shkel[1], Svenja Knappe[2], Elizabeth Donley[2], and John Kitching[2]
[1]Microsystems Laboratory, University of California, Irvine, CA, USA
[2]Time and Frequency Division, National Institute of Standards and Technology, Boulder, CO, USA

ABSTRACT

This paper presents an application of micro glass blowing, in which multiple glass spheres are simultaneously shaped on top of a silicon wafer and subsequently filled with rubidium. The fabrication process is based on etching cavities in silicon, followed by anodic bonding of a thin glass wafer to the etched silicon wafer. The bonded wafers are then heated inside a furnace at a temperature above the softening point of the glass, and due to expansion of the heated trapped gas in the cavities, the glass is blown into three-dimensional spherical cells. Microscopic alkali vapor cells are achieved by evaporation of ^{87}Rb through a small glass nozzle into the cell cavities. The cells are then sealed by anodic bonding. The results of the cell fabrication and characterization are presented.

1. INTRODUCTION

Recent advances in the field of atomic MEMS have created a need for microscopic alkali vapor cells [1-5]. While previous MEMS efforts have primarily been focused on flat vapor cells with only two optical ports [6-9], designed to be used in chip-scale atomic clocks (CSACs) and chip-scale atomic magnetometers (CSAMs) (see for example [1,2]), glass-blown spherical cells are traditionally used in macroscopic atomic clocks and other atomic apparatuses. Although glass blowing has been used to achieve millimeter-scale cells in the past [10,11], it has not previously been considered a viable option for mass production of microscopic devices.

It is well known from classical electrodynamics that the magnetic field inside a uniformly magnetized sphere is also uniform [12]. It is also known that self-fields in a non-spherical sample cause field variations across the sample and can lead to broadening of the magnetic resonance lines [13]. Spherical vapor cells are thus preferred in many atomic physics applications in order to minimize the effects of self-fields on the atoms. Furthermore, a spherical cell-shape provides more optical ports than a flat cell, which allows for straightforward pump-probe configurations, and enables vertical integration with other MEMS components. The presented micro glass blowing process was developed as an attempt to address these issues and to facilitate mass production of microscopic spherical gas confinement chambers.

In the wafer-level micro glass blowing process, thousands of glass spheres can be shaped simultaneously on top of a silicon wafer [14-16]. The fabrication process is based on etching cavities in silicon, followed by anodic bonding of a thin glass wafer to the etched silicon wafer. The bonded wafers are then heated inside a furnace at a temperature above the softening point of the glass, and due to expansion of the heated trapped gas in the cavities the glass is blown into three-dimensional spherical structures. Fig. 1 depicts a fabricated and diced chip containing a glass-blown sphere, formed during 3 minutes at 850°C. A close-up picture of a glass sphere with a diameter of approximately 900 μm is also shown.

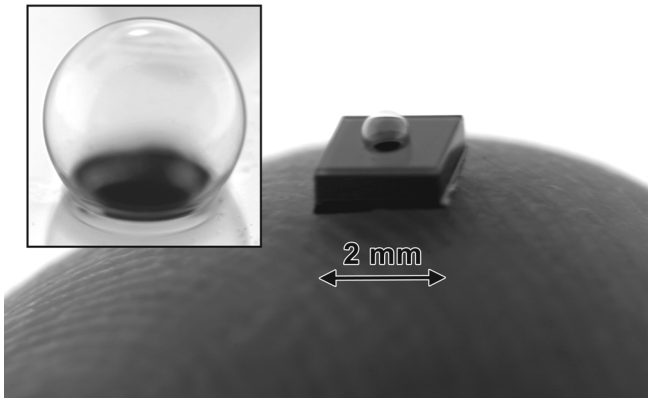

Figure 1: Diced silicon chip containing a glass-blown sphere (on a finger tip). The diameter of the glass sphere is approximately 900 μm.

The presented glass-blown vapor cells were originally developed for a micromachined implementation of a nuclear magnetic resonance gyroscope. However, the same type of cells may also be utilized in other microfabricated devices, such as atomic clocks and magnetometers.

We envision that micro glass blowing will open the door for a new breed of three-dimensional MEMS. In addition to the presented gas confinement chambers, several other novel applications are envisioned, including mass-produced microscopic glass lenses, spacers for wafer-level packaging, and complex three-dimensional networks for gas analyzers and drug delivery systems.

2. MEMS VAPOR CELLS

MEMS alkali vapor cells have previously been fabricated by etching a double-side polished silicon wafer all the way through, followed by anodic bonding of glass wafers to the top and bottom surfaces of the silicon wafer. The cells are filled with the desired substances before the second glass wafer is bonded. These types of vapor cells are an integral part of both chip-scale atomic clocks [1] and chip-scale atomic magnetometers [2]. While the fabrication of the glass-blown vapor cells presented in this paper differs from the CSAC and CSAM cells, the filling techniques are nearly identical.

Different filling techniques have been demonstrated for CSAC and CSAM cells [6-9]. Many of them can also be used to fill the spherical glass cells, fabricated by micro glass blowing. Here, we choose a filling technique where BaN_6 and $^{87}RbCl$ are placed inside a small glass ampoule with a 5 mm long nozzle of 700 µm diameter. The ampoule is then aligned with the cell opening inside a vacuum chamber and heated in order to react the compounds, and the nitrogen produced during the reaction is pumped away. Since the vapor pressure of the rubidium is higher than that of Ba and Cl, a fairly pure ^{87}Rb beam emerges from the ampoule and is deposited into the cell. The vacuum chamber is then filled with the desired combination of buffer gases and the cell is sealed by anodic bonding of a Pyrex wafer to the silicon.

3. MICRO GLASS BLOWING

In conventional glass blowing, a gob of glass is first heated inside a furnace. The gob is then removed from the furnace and blown into desired shapes. Often the heating and blowing steps are repeated multiple times. Once the glass is shaped, it is usually annealed to remove stresses that developed during the blowing.

The wafer-level micro glass blowing process was designed to emulate conventional glass blowing. However, instead of applying pressure by manual blowing, the increased pressure of a heated trapped gas is utilized to shape the glass. While an overview of the fabrication process is presented in this section, a more detailed description and modeling can be found in [16].

Fabrication Process

In the first fabrication step, a silicon wafer is patterned with a layer of AZ P4620 photoresist, as illustrated in Fig. 2. Cylindrical cavities are then etched in the silicon wafer using timed deep reactive ion etching (DRIE). The photoresist is removed with acetone and a 100 µm thin borosilicate glass wafer (Pyrex 7740) is anodically bonded to the silicon wafer in air, covering all of the etched cavities. Once bonded, the glass wafer may also be grinded and polished if thinner glass walls are desired.

The viscosity of glass is highly dependent on the temperature. At room temperature glass essentially behaves like an elastic solid, responding rapidly to applied stress. However, at sufficiently high temperatures, stress is immediately relieved from the material due to the low viscosity of heated glass. In order to shape glass, it needs to be heated above its softening point, which occurs at 821°C for the Pyrex 7740 glass used in the process described in this paper [17]. Therefore, in Step 3 of the fabrication process, the bonded wafers are placed inside a furnace at atmospheric pressure and at a temperature in the range of 850°C to 900°C. Due to the high temperature, the pressure of the trapped air inside the sealed cavities increases and the glass deforms into spherical shapes. After a few minutes in the furnace the samples are quickly removed and brought out to room temperature.

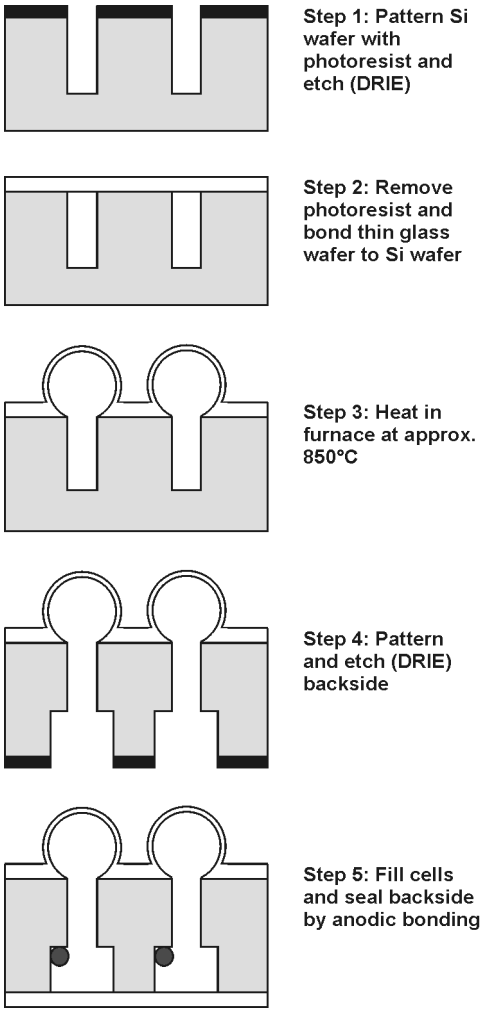

Figure 2: Wafer-level fabrication of rubidium vapor cells by micro glass blowing.

The rapid cool-down of the samples inevitably leads to residual stresses in the glass spheres. In order to remove these stresses, an annealing step can be added to the fabrication process. The glass should be annealed for about 30 minutes followed by a very slow cool-down to a temperature below the strain point of the glass. The annealing and strain points of Pyrex 7740 are 560°C and 510°C, respectively [17].

Fabrication Steps 1-3 in Fig. 2 constitute the foundation of the micro glass blowing process and define the shape and size of the glass structures. While these steps are always included in the fabrication process, additional steps can be added to suit a particular application.

In the gas confinement chambers explored in this paper, it is necessary to etch the backside of the silicon wafers in order to fill the glass spheres with an alkali metal and buffer gas. Once the backside is etched (Step 4), an aluminum holder is placed on top of the chip in order to protect the glass spheres. The cells are then filled with ^{87}Rb as well as a buffer gas mixture consisting of Xe and N_2 using a filling method previously utilized for CSACs and

CSAMs, discussed in Section 2. In the last fabrication step the backside is sealed by anodic bonding of a Pyrex wafer.

Size Estimation

As was previously described, the glass blowing takes place inside a furnace at atmospheric pressure. When the samples are placed inside the furnace, the high temperature will cause the pressure to increase rapidly inside the sealed cavities of the silicon wafer. Once the glass temperature reaches the softening point, the increased pressure inside the sealed cavities will force the glass to deform. The glass will assume a spherical shape and will continue to deform until the pressure inside the glass spheres is almost equal to the atmospheric pressure inside the furnace. At this point, most of the stress in the glass structures will have been relieved and the blown samples can be removed from the furnace. Since the final pressure is approximately equal on the inside and the outside of the glass spheres (before the samples are removed from the furnace), the ideal gas law can be used to estimate the final size of the glass spheres.

Assume that the cavities etched in silicon were sealed at room temperature. Also assume that the chips were placed in a furnace set to 900°C and at constant atmospheric pressure. Since the temperature inside the furnace is approximately 4 times higher (1200 K vs. 300 K) than the temperature at which the cavities were sealed, the total enclosed volume (glass sphere + etched cavity) after the glass is blown will be about 4 times the initial volume (etched cavity). Consequently, the final volume enclosed by only the glass sphere will be 3 times the volume of the etched cavity. Geometry considerations can be used to predict the height and radius of the glass spheres [16].

Fabricated Parts

2-inch diameter single-crystal silicon and Pyrex 7740 wafers were used for the fabrication of the glass spheres. An array of cylindrical cavities was first etched in the silicon wafer using deep reactive ion etching (DRIE). Structures have been successfully fabricated for etched diameters ranging from 100 µm to 1 mm. The targeted depth of the etched cavities varied from 300 µm to 800 µm.

After etching cavities in the silicon, a thin Pyrex 7740 wafer was anodically bonded to the silicon wafer. The bonding was done at atmospheric pressure on top of a hot plate set to 400°C and using a voltage of 600 V. The samples were then placed inside a furnace at 850°C and at atmospheric pressure for about 3 minutes. Fig. 3 shows a fabricated array of glass spheres.

In order to be able to perform metrology, the fabricated glass structures were covered with photoresist and diced at the center of the spheres. A scanning electron microscope (SEM) image of the cross-section of one of the hollow glass spheres is shown in Fig. 4 [16]. The sample was fabricated using a 1 mm thick silicon wafer bonded to a 100 µm thin Pyrex 7740 wafer. The cylinder-shaped etched cavity was 750 µm deep and 500 µm in diameter, and the diameter of the glass sphere was approximately 900 µm.

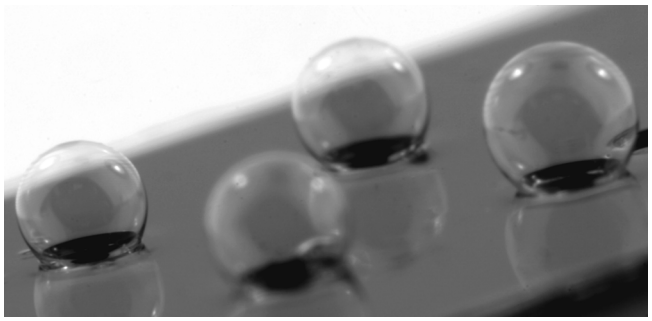

Figure 3: Fabricated array of micro glass spheres.

The thickness of the glass sphere was approximately 7 µm at the horizontal top surface and 16 µm at the vertical side surface. In all samples that were diced and measured, the thickness of the top ranged between 40% and 60% of the thickness of the side. The average surface roughness of the dashed area in Fig. 4 was measured to be 2 nm on the inside and 9 nm on the outside [16], which is a slight increase from the specified Pyrex roughness of <10 Å.

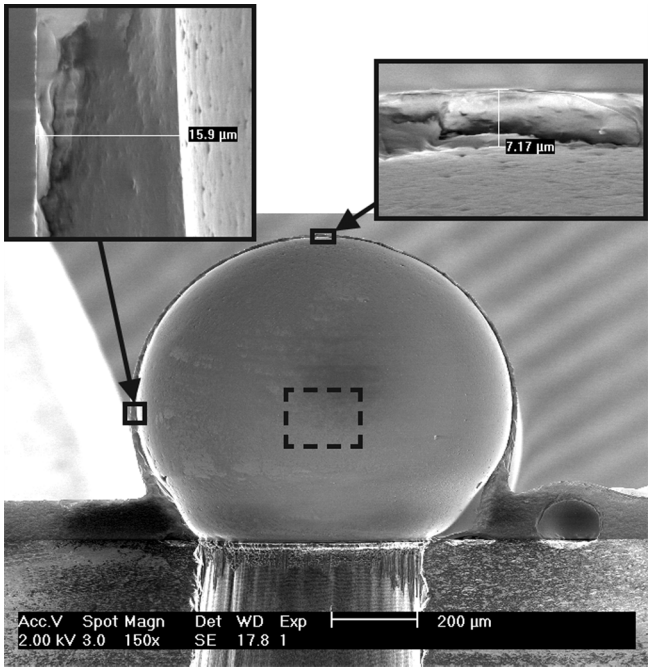

Figure 4: SEM image of a cross-section of a micro glass sphere [16].

4. EXPERIMENTAL RESULTS

Fig. 5 shows the experimental setup that was used to characterize the rubidium vapor cells. The light source was a vertical-cavity surface-emitting laser (VCSEL) with a thermoelectric cooler (TEC), which was used to control the VCSEL temperature. The light was collimated and transmitted through the rubidium vapor cell, and a photodetector was used to measure the intensity of the transmitted light. In order to obtain sufficient rubidium

vapor pressure inside the cell, it was heated to 100°C. Note that the Helmholtz coils in the experimental setup were not used in the current experiment.

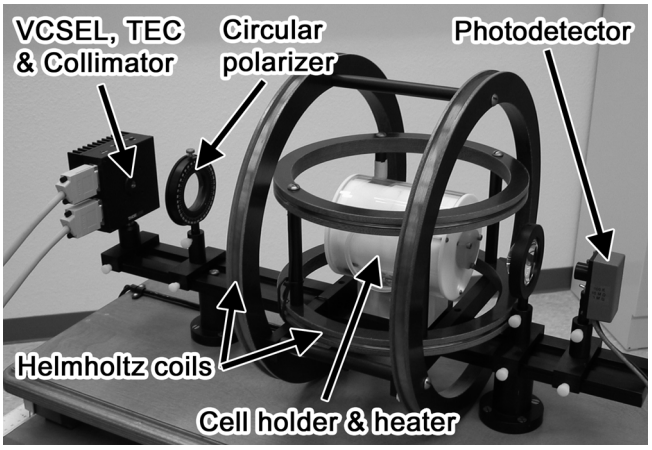

Figure 5: Experimental setup.

The VCSEL current was swept from 1.65 mA to 1.75 mA, corresponding to wavelengths of approximately 794.72 nm to 794.80 nm. Fig. 6 shows the obtained rubidium absorption spectrum. At the wavelength that corresponds to the ^{87}Rb D_1 line (794.76 nm in air) the light is partially absorbed by the rubidium and the intensity of the transmitted light drops, confirming that the cell contains ^{87}Rb vapor. The two overlapped peaks, separated by 6.8 GHz, arise from the hyperfine ground states of ^{87}Rb. This plot verifies the feasibility of the spherical vapor cells, fabricated by a wafer-level glass blowing process.

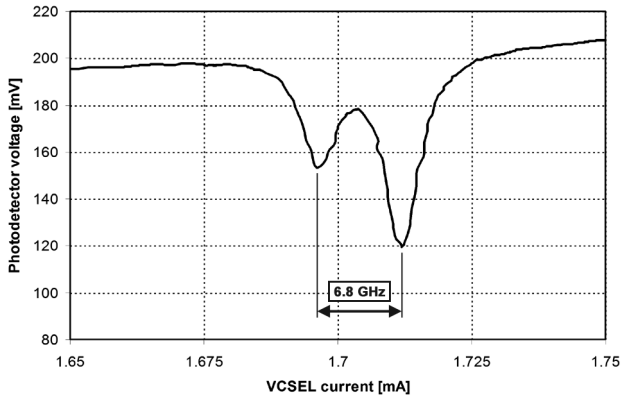

Figure 6: ^{87}Rb absorption spectrum for VCSEL currents of 1.65 mA (~794.72 nm) to 1.75 mA (~794.80 nm).

5. CONCLUSION

Wafer-level micro glass blowing may enable several novel MEMS applications. One potential application, glass blowing of microscopic gas confinement chambers, was explored in this paper. The feasibility of glass-blown spherical rubidium vapor cells was experimentally verified.

ACKNOWLEDGEMENTS

The authors would like to thank the staff of the UCI microfabrication facility, INRF, for fabrication support and Dr. Dragos Constantin for technical assistance. This work is supported in part by the DARPA NGIMG program.

REFERENCES

[1] S. Knappe, V. Shah, P. Schwindt, L. Hollberg, J. Kitching, L. Liew, and J. Moreland, "A microfabricated atomic clock," *Appl. Phys. Lett.*, vol. 85, pp. 1460-1462, 2004.

[2] P. Schwindt, S. Knappe, V. Shah, L. Hollberg, J. Kitching, L. Liew and J. Moreland "Chip-scale atomic magnetometer," *Appl. Phys. Lett.*, vol. 85, pp. 6409-6411, 2004.

[3] R. Lutwak, D. Emmons, T. English, W. Riley, A. Duwel, M. Varghese, D. K. Serkland, and G. M. Peake, "The chip-scale atomic clock – recent development progress," *Proc. 35th Ann. Precise Time and Time Interval Meeting*, pp. 467-478, 2003.

[4] M. Zhu, L. S. Cutler, J. E. Berberian, J. F. DeNatale, P. A. Stupar, and C. Tsai, "Narrow linewidth CPT signal in small vapor cells for chip scale atomic clocks," *Proc. IEEE Intl. Freq. Cont. Symp.*, pp. 100-103, 2004.

[5] D. W. Youngner, J. F. Detry, and J. D. Zook, "MEMS frequency standard for devices such as atomic clock," U.S. Patent 6,900,702, 2005.

[6] L. A. Liew, S. Knappe, J. Moreland, H. Robinson, L. Hollberg, and J. Kitching, "Microfabricated alkali atom vapor cells," *Appl. Phys. Lett.*, vol. 84, pp. 2694-2696, 2004.

[7] C.-H. Lee, H. Guo, S. Radhakrishnam, A. Lal, C. Szekely, T. McClelland, and A. P. Pisano, "A batch fabricated rubidium-vapor resonance cell for chip-scale atomic clocks," *Proc. Solid State Sensors, Actuators and Microsystems Workshop*, Hilton Head Island, SC, 2004.

[8] F. Gong, Y.-Y. Jau, K. Jensen, and W. Happer, "Electrolytic fabrication of atomic clock cells," *Rev. Sci. Instrum.*, vol. 77, 076101, 2006.

[9] S. Knappe, V. Gerginov, P. D. D. Schwindt, V. Shah, H. G. Robinson, L. Hollberg, and J. Kitching, "Atomic vapor cells for chip-scale atomic clocks with improved long-term frequency stability," *Opt. Lett.*, vol. 30, pp. 2351-2353, 2005.

[10] S. Knappe, V. Velichansky, H. G. Robinson, J. Kitching, and L. Hollberg, "Compact atomic vapor cells fabricated by laser-induced heating of hollow-core glass fibers," *Rev. Sci. Instrum.*, vol. 74, pp. 3142-3145, 2003.

[11] M. V. Balabas, D. Budker, J. Kitching, P. D. D. Schwindt, and J. E. Stalnaker, "Magnetometry with millimeter-scale antirelaxation-coated alkali-metal vapor cells," *J. Opt. Soc. Am. B*, vol. 23, pp. 1001-1006, 2006.

[12] J. D. Jackson, *Classical Electrodynamics,* 3rd ed. New York: Wiley, 1999.

[13] C. Kittel, *Introduction to Solid State Physics,* 8th ed. New York: Wiley, 2005.

[14] E. J. Eklund and A. M. Shkel, "Wafer-level micro-glass-blowing," *UCI Office of Technology Alliances*, UC Case No. 2006-176, 2005.

[15] E. J. Eklund and A. M. Shkel, "Glass blowing on a wafer scale (expanded version of 2006-176)," *UCI Office of Technology Alliances*, UC Case No. 2006-519, 2006.

[16] E. J. Eklund and A. M. Shkel, "Glass blowing on a wafer level," *J. Microelectromech. Syst.*, Submitted.

[17] Corning, "Thermal properties of Corning glasses," 2006. [Online]. Available: http://www.corning.com/lifesciences/technical_information/techdocs/thermalprop.asp.