

MICROMACHINED ALKALI ATOM VAPOR CELLS FOR CHIP-SCALE ATOMIC CLOCKS

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ABSTRACT

This paper describes the fabrication of chip-scale alkali atom vapor cells, for use in highly miniaturized atomic frequency references, using silicon micromachining and anodic bonding technology. The cells consist of silicon cavities with internal volume ranging from a few mm³ to less than 1 mm³. The cells were filled with cesium and nitrogen buffer gas either by chemical reaction of cesium chloride and barium azide, or by direct injection of elemental cesium within a controlled anaerobic environment. Cesium optical absorption spectra were obtained from the cells, and coherent population trapping resonances with linewidths of about 1 kHz were measured.

1. INTRODUCTION

There has been much recent interest in highly miniaturized atomic clocks for various military and civilian applications such as synchronization of encryption keys, GPS signals, and communications networks. This paper discusses the first micromachined alkali atom vapor cells, which is a significant step toward realizing miniaturized, chip-scale atomic clocks (CSACs).

Atomic clocks are based on transitions between the ground-state hyperfine levels of an alkali atom, which is usually cesium (Cs) or rubidium (Rb). The heart of an atomic clock is its "physics package" which consists of the cesium or rubidium atoms and the means to excite them and detect their transitions. The physics packages of commercial small atomic clocks [1] are typically several cm³ in volume.

In conventional atomic clocks, a microwave field from an external oscillator is applied to the atoms and the frequency of the oscillator is stabilized with respect to the atomic transitions in the atoms [2]. However, there have been difficulties in making resonant cavities with dimensions smaller than the microwave wavelength in air; thus, size reduction is limited to the centimeter scale when using this conventional method of exciting the atoms.

Alternative excitation techniques have thus been investigated, including coherent population trapping (CPT) resonances. Recently, Kitching et al [3], [4] demonstrated a compact atomic clock based on all-optical CPT excitation, in which two optical fields – obtained by modulating a single field from a vertical cavity surface emitting laser (VCSEL) to create sidebands – are applied to a cesium vapor cell. At a critical frequency difference between the two fields, a dark line resonance or CPT resonance occurs, signified by a change in the cesium atoms' optical absorption. This resonance is then used to lock the modulation frequency of the laser. The advantage of using this CPT method over

conventional microwave excitation is that the physics package can be made much smaller and simpler. Our aim, therefore, is to develop a CSAC physics package based on the CPT scheme, and with volume less than 3 mm³.

Optically transparent cells containing cesium or rubidium and a buffer gas are an important clock component. Hence, a first milestone toward realizing CSACs is to develop fabrication processes for highly miniaturized cells with volume less than 3 mm³. Traditional glass-blowing techniques with gas torches have conventionally been used to make atomic vapor cells; however, these cell fabrication methods are not feasible at sizes below 1 cm. Recently, cells with an inner volume of 5 mm³ were fabricated by CO₂ laser-induced heating of hollow-core glass fibers and appears promising [5].

The approach taken in this work was to use a completely different technology, silicon micromachining, to fabricate small cells. In addition to the gains in miniaturization, other advantages of silicon micromachining over glass-blowing technology include lower-cost, batch fabrication, and the use of the same manufacturing platform as found in microelectronics and Micro-electro-mechanical-systems (MEMS), thus making possible monolithic and hybrid integration with control electronics and sensors.

Cesium is the most reactive of the alkali metals and cannot be handled in air, thus while many micromachining techniques such as silicon etching and silicon-to-glass anodic bonding are well known, a significant challenge associated with developing chip-scale vapor cells lies in introducing cesium or rubidium and a buffer gas into a micromachined cell cavity and hermetically sealing the cavity without the need for microvalves and/or glass tubing connections. (In addition to the alkali atoms, the type of buffer gas and its pressure are important for clock applications.) These challenges have not, to our knowledge, been previously encountered in applications utilizing silicon micromachining.

2. EXPERIMENTAL METHODS

Silicon and Corning 7740 (PyrexTM) [6] were the chosen materials with which to fabricate the cells, silicon being easy to micromachine and glass being optically transparent for CPT interrogation and having well known reactive properties with alkali atoms. Furthermore, silicon-to-glass anodic bonding is a well-characterized process. Figure 1 shows a schematic of the overall cell structure and fabrication process. Double-sided polished <100> silicon wafers (a) were photolithographically patterned and etched using either KOH or deep reactive ion etching to produce through-holes of sizes ranging from a few millimeters to 100 microns, and were then diced into individual chips of roughly 1 cm in length and width (b); alternatively, this separation may be

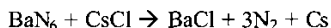
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accomplished as part of the etching. The silicon chips were then anodically bonded to similar-sized pieces of Pyrex (c) by heating on a hotplate to about 300 °C and applying 1000 volts across the silicon-Pyrex interface. We refer to the resulting silicon-glass structure as a “preform”.

The preform cavities were then filled with cesium and buffer gas (which in our experiments was N₂) (d), and the cell cavity was sealed by anodically bonding a second Pyrex chip to the top free surface of the silicon preform (e). The final cell consisted of a 3-layered bonded structure (f), with optically-transparent Pyrex windows on either side of the cesium-containing silicon cavity. Steps (d) and (e) are now described.

We have developed two methods for filling and sealing the micromachined cells: (1) using the chemical reaction between barium azide and cesium chloride within an ultra-high vacuum (UHV) system, and (2) direct-injection of liquid cesium within a low-vacuum anaerobic chamber.

In the chemical reaction approach a stable cesium compound is introduced into the preform cavity and subsequently reacted to obtain pure Cs. CsCl salt was added to a 15% solution of BaN₆ in H₂O to produce a clear colorless liquid which was then introduced into the preform cavities using a microliter pipette. The preform was baked to evaporate the water, leaving a white solid residue inside the cavity. The preform was then placed in a custom ultra high vacuum (UHV) chamber containing heaters, positioning mechanisms, and electrodes for anodic bonding. The chamber was evacuated to a pressure of about 10⁻⁴ Pascals and the preform was heated to 120 °C, causing the BaN₆ to decompose into N₂ and elemental Ba. This N₂ gas was removed by further evacuating the chamber. The UHV chamber was then backfilled with the desired pressure of N₂ buffer gas. (Alternatively, other buffer gases or gas mixtures could be backfilled.) A Pyrex chip was then pushed against the preform’s top surface and anodically bonded to seal the cell. Finally, when the preform was heated to around 200 °C the Ba reacted with the CsCl to produce BaCl and elemental Cs. The reaction is summarized as follows [7]:



Thus, elemental Cs was produced inside the sealed preform cavity, along with BaCl which is a white solid, and nitrogen buffer gas.

Since an ultra-high vacuum environment is not in and of itself necessary for cell fabrication except to produce a non-oxidizing atmosphere for handling cesium, an alternative approach to handling cesium in an oxygen-free environment is by using a commercial anaerobic chamber. This glove box contains predominantly dry nitrogen but also an anaerobic gas mixture which, through catalytic reaction, removes trace amounts of oxygen by reacting with it to form water vapor which is then absorbed by the system’s drying train. This method of producing a controlled oxygen- and moisture-free environment is well-documented and used in industry and in microbiology research. Within the glove box is a custom low-vacuum bell-jar containing fixtures, heaters and electrodes for anodic bonding.

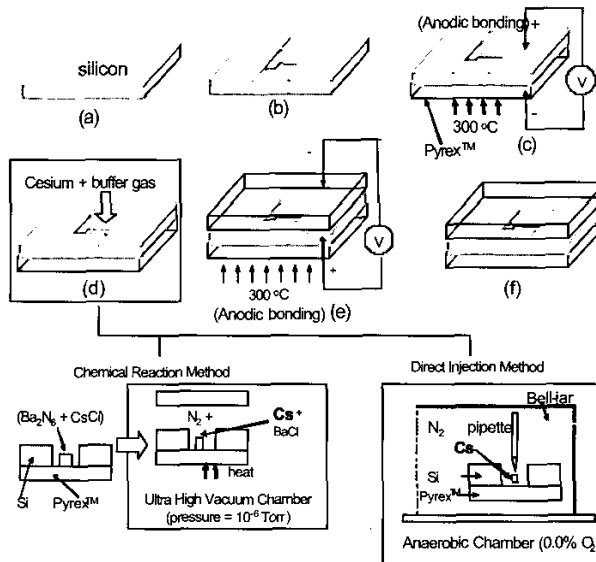


Figure 1. Schematic of cell fabrication process.

In these experiments, the anaerobic chamber was thoroughly purged with dry nitrogen and the catalytic reaction/drying system was operated to obtain an internal glove box atmosphere containing less than 0.1% oxygen (as measured using a galvanic sensor) and about 14% relative humidity at room temperature. It is believed that a further reduction in relative humidity to 1% or less may be achieved with this same commercial chamber through design improvements. Within the anaerobic glove box, a pipette was used to dispense 500 nanoliters of liquid cesium from a commercially-obtained ampoule into a preform cavity. The preform was then placed in the bell-jar fixture with a Pyrex chip in contact with the top silicon surface. The bell-jar was evacuated to a pressure of about 1 kPa, subsequently backfilled with N₂ buffer gas to the desired pressure of 20 – 35 kPa, and the pyrex chip was anodically bonded to the preform by heating to about 250 °C and applying 1000 V. Given the great affinity of alkali metals for oxygen and the high surface-to-volume ratio of these minute quantities of cesium, minimizing the cesium oxidation is a challenge, and most cells produced this way contain some white cesium oxide in addition to pure cesium. The chemical reaction method, on the other hand, did not exhibit such oxidation limitations. The optical transparency of the glass windows could be compromised by the presence of either the cesium oxide (direct injection method) or barium chloride (chemical reaction method). Possible solutions to this include fabricating separate cavities in the silicon as filling reservoirs for either the cesium or the chemical precursors, thus separating the oxides or byproducts from the main optical window. Alternatively, an atomic beam of alkali atoms in a UHV environment could be used to deposit cesium cleanly into a cell before bonding.

Figure 2 shows a typical micromachined cell. The cesium may be visually identified as shiny metallic-looking particles in the cell window. The smallest cells we have made have cavities of dimension 0.75 mm x 0.75 mm x 0.35 mm.

In principle the entire fabrication and filling process may be performed at the wafer level with subsequent dicing into individual cells. Furthermore, pressure and temperature sensors may be readily fabricated into the cell itself as additional micromachining processes, leading to cells with increased functionality and a further decrease in the overall size of the physics package.

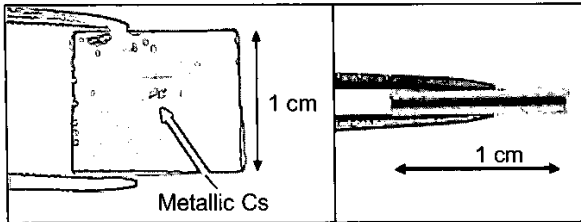


Figure 2. Photograph of the top view (left) and side view (right) of a typical micromachined cell.

3. EXPERIMENTAL RESULTS

Optical Absorption Resonance

Optical absorption measurements were performed to confirm the presence of both cesium and the buffer gas in the cells. The cells were heated to a temperature between 55 °C and 80 °C, light from a VCSEL was passed through the windows and the wavelength was scanned over the D2 transition in cesium at 852 nm. Figure 3 shows the typical cesium optical absorption consisting of two resonances separated by 9.2 GHz corresponding to the two ground state hyperfine levels. As expected, Doppler broadening is seen in the absorption lines with increasing buffer gas pressure. It has been found that the optimum pressure for nitrogen buffer gas is 20 – 35 kPa. Excessively higher buffer gas pressures result in overly broadened optical transitions and may render the cell difficult to use in clock applications. At the other extreme, too little buffer gas results in increased collisions between the cesium atoms and the cell walls, reducing the Q and thus the cell's effectiveness in a clock. Of the two spectra in Figure 3, (b) indicates a cell more suitable for a small clock application because its buffer gas pressure lies within the desired range, as determined by the broadening of the optical transition.

CPT Resonance

Figure 4 shows CPT resonances for cells made using both filling methods. The cell made using the chemical reaction method [trace (b)] is slightly larger than that made using the direct injection method [trace (a)], and has a narrower linewidth. Under shot-noise-limited conditions, a resonance with the width and contrast of trace (a) should result in a short-term frequency instability of about $2 \times 10^{-10} / \sqrt{\tau}$.

We also demonstrate narrower CPT resonances from these micromachined cells. In general, narrow resonances lead to better stability and higher signal-to-noise ratio in the final clock. We chose a 1 kHz FWHM linewidth

as a rigorous benchmark to assess the capability of our cell fabrication process.

Sub-kHz resonances fundamentally require a cell volume on the order of about 10 mm³. The internal dimensions of the cell cavity in this case were 2 mm x 2 mm x 1.8 mm thick. To obtain such large cell volumes from our micromachining process, commercially-obtained 1mm-thick silicon wafers were etched with through-holes of lateral dimensions 2 mm x 2 mm. Two of these thick etched silicon chips were then anodically bonded to either side of a Pyrex chip creating a Si/glass/Si sandwich structure. A through-hole was etched in the Pyrex using a hydrofluoric acid bath with the bonded silicon layers as etch masks. Another Pyrex layer was then bonded to the bottom of the stack to create the preform. In this way, preforms were fabricated with silicon/glass/silicon cavities of optical path length 1.8 to 2.5 mm, depending on the thickness of the pyrex and/or silicon wafers. These preforms were then filled with Cs and buffer gas and sealed as described.

Cells with sub-kHz linewidths were successfully fabricated with nitrogen buffer gas pressure of 29 kPa, as shown in Figure 5. Even though a sub-kHz linewidth is not critical for clock operation, this study shows that our fabrication processes are capable of producing cells with high Q.

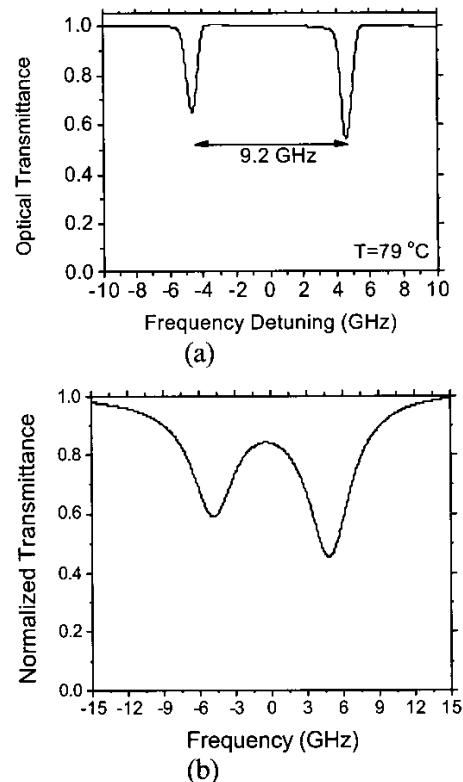


Figure 3. Typical optical absorption spectrum of cesium cells, showing the effect of line broadening due to an increase in buffer gas pressure. The cell in (a) has a buffer gas pressure of about 1 kPa while the cell in (b) has a buffer gas pressure of 29 kPa. In both cases the buffer gas is N₂.

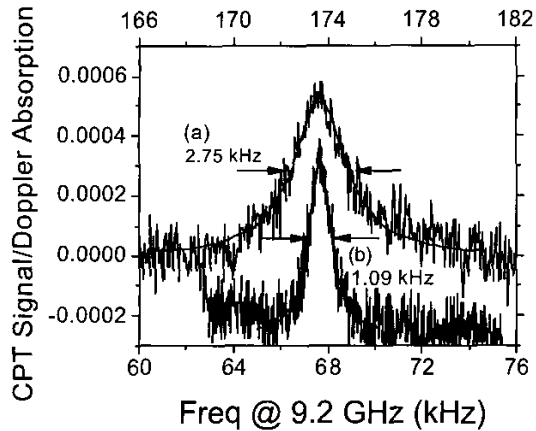


Figure 4. CPT resonances measured in micromachined cesium cells. Trace (a), [lower x-axis scale] corresponds to a cell made using the direct injection method, trace (b) [upper x-axis scale] is for a cell made using the chemical reaction method. Cell (a) had inner dimensions of 2 mm x 2 mm x 1 mm and a temperature of 50°C. Cell (b) had inner dimensions of 2 mm length and 1.25 mm radius and a temperature of 85°C. Trace (b) has been offset by 0.0003 for clarity.

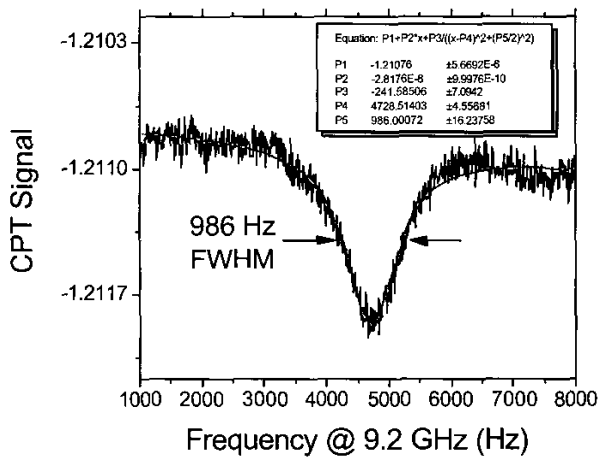


Figure 5. CPT resonance with a sub-kHz linewidth, measured in a micromachined cesium vapor cell. The equation of the Lorentzian fit is shown.

4. CONCLUSION

We have developed processes to fabricate chip-sized cesium vapor cells using silicon micromachining technology. Optical absorption and CPT resonances were obtained, indicating the suitability of these cells for atomic clock applications. In addition, CPT linewidths of 1 kHz or less were measured in these cells. Thus, an important first step toward realizing chip-scale atomic clocks has been accomplished.

ACKNOWLEDGEMENTS

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