Microfabricated alkali atom vapor cells

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We describe the fabrication of chip-sized alkali atom vapor cells using silicon micromachining and anodic bonding technology. Such cells may find use in highly miniaturized atomic frequency references or magnetometers. The cells consist of cavities etched in silicon, with internal volumes as small as 1 mm^3. Two techniques for introducing cesium and a buffer gas into the cells are described: one based on chemical reaction between cesium chloride and barium azide, and the other based on direct injection of elemental cesium within a controlled anaerobic environment. Cesium optical absorption and coherent population trapping resonances were measured in the cells. [DOI: 10.1063/1.1691490]

We describe the fabrication of micromachined chip-sized alkali atom vapor cells. Such cells may find use in applications such as highly miniaturized magnetometers, spectroscopic reference cells, and atomic frequency references. In particular, there has been much recent interest in highly miniaturized atomic clocks (volume <1 cm^3) for various military and civilian applications, including antijam global positioning, and synchronization of encryption keys and communications networks. The atoms commonly used as frequency standards are H, Rb, and Cs. The size reduction of physics packages based on conventional microwave excitation of these atoms has been limited to the centimeter scale due to difficulties in making microwave resonant cavities with dimensions smaller than the microwave wavelength. Alternative atom excitation techniques have thus been investigated, including coherent population trapping (CPT) resonances.[1] Recently, a compact atomic clock based on all-optical CPT excitation was demonstrated,[2,3] 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 Cs 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 chip-scale (about 3 mm^3) physics package for an atomic clock based on CPT excitation.

An important milestone toward realizing an ultrasmall physics package is the development of methods to fabricate highly miniaturized atomic vapor cells with volumes on the order of 1 mm^3 or less. 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 inner volume 5 mm^3 were fabricated by CO_2 laser-induced heating of hollow-core glass fibers,[4] and this technique appears promising for realizing millimeter-sized cells.

The approach taken in this work was to use a completely different technology—silicon micromachining—to fabricate small cells. In addition to allowing a higher level of miniaturization, other advantages of using silicon micromachining over glass-blowing technology include lower cost and higher reproducibility afforded by wafer-level batch fabrication, and the use of the same manufacturing platform as microelectronics and microelectromechanical systems, thus making possible the monolithic and hybrid integration with control electronics and sensors. Because cesium reacts with many materials and cannot be handled in air, the objective in addition to fabricating small cell cavities was to develop processes to introduce cesium or rubidium and a buffer gas into the cell without the need for microvalves and/or glass tubing connections, and to effectively seal this environment within the cell. (In addition to the alkali atoms, the buffer gas type and pressure is important for certain applications such as clocks.)

Silicon and Corning 7740 (Pyrex™)[5] 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[6] 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 in a cleanroom and etched in either KOH or using deep reactive ion etching to produce
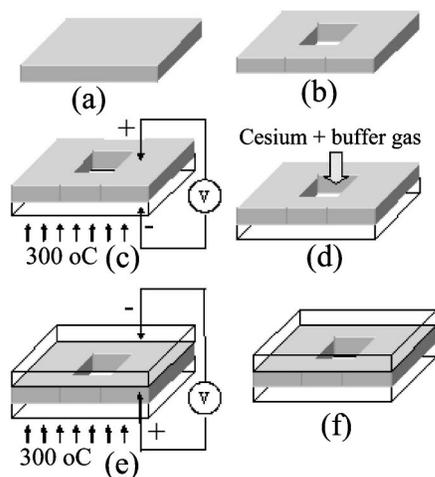
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FIG. 1. Schematic of cell fabrication based on silicon etching and anodic bonding.

FIG. 2. (a) Photograph of the top view of a cell showing the cesium contained within. (b) Photograph of a smaller cesium cell.

square through-holes of sizes ranging from a few millimeters to 100 μm; alternatively, larger-sized circular holes were made by drilling. The silicon wafers were then diced into individual chips of roughly 1 cm in length and width (b); alternatively, this separation may be accomplished as part of the etching. The silicon chips were then anodically bonded to similarly-sized pieces of Pyrex (c) by heating on a hotplate to about 300 °C and applying 1000 V across the silicon-pyrex interface with the cathode in contact with the Pyrex. The combination of heat and high electric field caused oxides to form at the interface, thereby creating a hermetic bond between the silicon and Pyrex. We refer to the resulting bonded stack as a "preform."

The preform cavities were then filled with cesium and a nitrogen buffer gas (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 three-layered bonded structure (f), with optically transparent Pyrex windows on either side of the cesium-containing cavity. Steps (d) and (e) are now described in detail.

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 UHV system followed by anodic bonding, and (2) direct injection of liquid cesium within a low-vacuum anaerobic chamber followed by anodic bonding.

In the chemical reaction approach, a stable cesium compound is introduced into the preform cavity and subsequently reacted to obtain pure cesium. CsCl salt was added to a 15% solution of BaN_6 in H_2O 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 UHV chamber containing heaters and electrodes for anodic bonding. The chamber was evacuated to a pressure of about 10^{-6} Torr and the preform was heated to 120 °C, causing the BaN_6 to decompose into N_2 and elemental Ba. A Pyrex chip was pushed against the preform's top surface, and the N_2 gas was removed by leaving the preform in the UHV system for at least an hour. The UHV chamber was then backfilled with the desired pressure of N_2 buffer gas. A voltage of 1 kV was then applied,
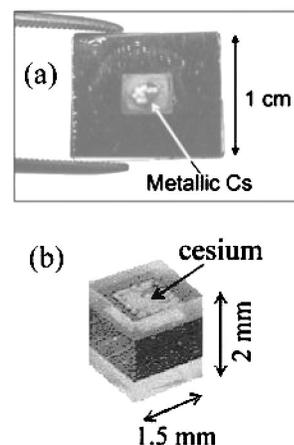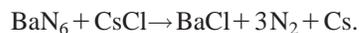
and the temperature was gradually increased to above 200 °C, initiating the anodic bond between the Pyrex chip and the preform and thus hermetically sealing the cavity. Finally, at around 200 °C, the Ba reacted with the CsCl to produce BaCl and elemental Cs. This reaction is summarized as follows:[7]

$$BaN_6 + CsCl \rightarrow BaCl + 3N_2 + Cs.$$

Thus, elemental cesium was produced inside the sealed preform cavity, along with barium chloride which is a white solid, and a dark colored solid that could be residual elemental barium, and nitrogen buffer gas. Note that the nitrogen produced from the chemical reaction itself was initially evacuated before the start of bonding, in order to avoid excessive buffer gas in the final cell. In addition, the initial evacuation of nitrogen allows us to backfill with any type of buffer gas or gas mixture at the desired pressures.

Since an UHV environment is not (in and of itself) necessary for cell fabrication except to produce a nonoxidizing atmosphere for handling cesium, an alternative approach is to achieve this oxygen-free environment by using a commercial anaerobic chamber filled with dry nitrogen. Trace amounts of oxygen ($<1\%$) inside the chamber were removed via catalytic reactions with an anaerobic gas mixture and a palladium catalyst. The catalytic reactions reduced the oxygen to form water vapor, which was then absorbed by the system's drying system. This method of producing a controlled, oxygen- and moisture-free environment is well documented, commonly used in industry and in microbiology fields, and is highly developed. In addition, such a chamber is essentially a glove box that allows direct manual manipulation of samples via gloves that extend into the chamber. Within the anaerobic chamber, a nanoliter pipette was used to dispense 500 nl of liquid cesium from an ampoule into the preform cavity. The preform was then placed inside a custom low-vacuum bell jar within the anaerobic chamber, and which contained heaters and electrodes for anodic bonding. A Pyrex chip was pressed against the preform, the bell jar was evacuated to a pressure of about 1 Torr and then subsequently backfilled with the anaerobic glove-box atmosphere (85% N_2,10% H_2,5% CO_2) to the desired pressure of 150– 200 Torr. High voltage (1000 V) was then applied as the preform was heated to 250 °C to bond the Pyrex to the pre-
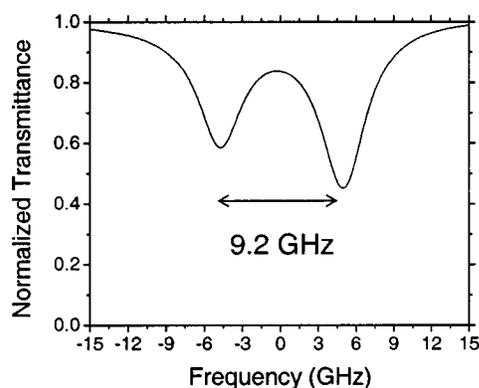
FIG. 3. Typical optical absorption spectrum of cesium cells made using direct injection of cesium in an anaerobic chamber. The broad absorption line indicates the presence of a substantial amount of buffer gas (about 20 kPa, assuming the buffer gas is N_2). Similar absorption spectra are seen for cells made using the chemical reaction method.

FIG. 4. CPT resonances from two cells, one made using the direct injection method [trace (a), lower *x*-axis scale] and the other made using the chemical reaction method [trace (b), upper *x*-axis scale]. Cell (a) had inner dimensions of 2 mm×2 mm×1 mm and temperature of 50 °C. Cell (b) had inner dimensions of 2 mm length and 1.25 mm radius and temperature of 85 °C. Trace (b) has been offset by 0.0003 for clarity.

form, sealing the cesium and buffer gas inside the cell. The cesium can be visually identified as shiny, metallic-looking particles inside the cell. Figure 2 shows cells made using both filling methods described.

Optical absorption measurements were performed to confirm the presence of cesium and buffer gas in the cells made by both methods. The cells were heated to a temperature between 55 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. The resulting spectra in Fig. 3 show the typical cesium optical absorption consisting of two resonances separated by 9.2 GHz, corresponding to the two ground state hyperfine levels. 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 deposition 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 $2 \times 10^{-10}/\sqrt{\tau}$.

The pressure of the buffer gas in the cells can be estimated from either the broadening of the optical transition (Fig. 3) or the frequency shift of the CPT resonance (Fig. 4). For the cell made using the chemical reaction technique, the two estimates were consistent and yielded a value of 29 kPa. For this technique, we estimate that the uncertainty in obtaining a prespecified buffer-gas pressure is near 20%, limited mostly by the uncertainty in the measurement of the preform temperature during bonding. For the cell made by the direct injection technique, the estimate based on the CPT frequency shift is complicated by the mixture of buffer gases in the cell. However, from the optical broadening data, it is possible to deduce that the total pressure is near 20 kPa, which is roughly what was intended. In future, the gas composition could be controlled by connecting a separate gas line to the bonding chamber for the backfilling step, instead of backfill-
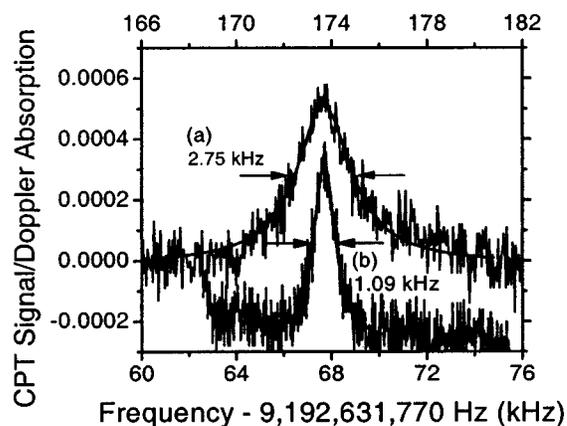
ing the bonding chamber with the anaerobic chamber's atmosphere. Nevertheless, we have been able to fabricate cells with buffer-gas pressures ranging from below a few tens of Pa to several hundred kPa. The wide range of possible buffer-gas pressures and the ability to fill the cell with a precise buffer-gas pressure make this cell fabrication technique suitable for a large number of clock and magnetometer applications. Both methods for cell filling and sealing studied in this work thus appear viable and have been used to obtain micromachined cesium vapor cells.

The entire cell fabrication, filling, and sealing may, in principle, be performed at the wafer level, with subsequent dicing into individual cells being the last step. 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.

We have developed methods to fabricate millimeter-sized cesium vapor cells—with the potential of further size reduction to submillimeter volumes—using silicon micromachining and anodic bonding techniques. Optical absorption and CPT resonances were obtained, indicating the suitability of these cells for use in highly miniaturized atomic frequency references based on an all-optical CPT excitation scheme.

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[5] Trade name is stated for technical clarity and does not imply endorsement by NIST. Products from other manufacturers may perform as well as or better.
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