

WAFER-LEVEL FABRICATION AND FILLING OF CESIUM-VAPOR RESONANCE CELLS FOR CHIP-SCALE ATOMIC DEVICES

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Abstract: We describe the wafer-level fabrication of cesium vapor cells by evaporation and photolysis of cesium azide for chip-scale atomic devices. The advantage is that, unlike existing cell-fabrication methods which require fluid handling devices for filling individual and separately fabricated cells, here the filling step itself is done by thin-film deposition and thus integrated monolithically with the rest of the fabrication process.

Keywords: atomic clock, vapor cell, cesium azide

INTRODUCTION

Microsystems technology has recently been applied to miniaturizing atomic clocks. These miniaturized atomic frequency references may find use in civilian and military applications such as global positioning and communications networks. Our group at NIST recently developed the first chip-scale atomic clock physics package [1], followed by other groups [2]. We have also developed a chip-scale atomic magnetometer [3], using similar principles and techniques. Thus, the application of MEMS to atomic systems has enabled a new class of chip-scale atomic devices (CSADs).

Central to these CSADs are the alkali metal vapor cells [4, 5] for containing cesium or rubidium vapor and buffer gases. In clocks, the frequency is based on transitions between the alkali atoms' ground-state hyperfine levels. In magnetometers, a magnetic field is measured based on the spin precession frequency of the atoms. Thus, alkali vapor resonance cells form the core of these atomic devices, and an ongoing challenge is to develop suitable techniques for fabricating chip-scale vapor cells such that they will not only be in a physical form suitable for integration into the rest of a MEMS-type assembly, but also in a manner that is amenable to true wafer-scale mass production.

Several groups [1 - 8] have recently developed techniques for cell microfabrication. Whereas conventional cells are made by glass blowing, those made using microfabrication technology most efficiently take the form of small holes or cavities that have been etched in silicon wafers and closed off by glass windows. These cells are easily fabricated using standard photolithography, silicon etching and bonding techniques in a cleanroom; however, *filling* the cells with the highly volatile alkali metal without exposure to air, and subsequent hermetic sealing of the cells, have posed an ongoing MEMS packaging challenge. Furthermore, the high temperature required for anodic bonding of silicon to glass (for hermeticity and to create glass windows for optical interrogation) is at odds with the low melting point of cesium and rubidium. Thus to date the existing

methods of cell fabrication have required technically complex methods to introduce the alkali metal into the microfabricated cell and to subsequently seal the cell hermetically.

Our previous approaches to cell filling involved pipetting liquid cesium into the cell cavity in an anaerobic glovebox [4], or reacting chemical precursors in an ampoule to produce the pure alkali metal, which is then diffused into the cell cavity in high vacuum [6]. Lee et al. [7] developed a method for filling cells using microchannels connected to a glass filling tube, followed by wax to seal the filling ports in the wafer. Radhakrishnan and Lal [8] took this work further by developing a method of encasing liquid rubidium in wax in a glovebox and using laser ablation to release the rubidium into the cell cavity. While each method has its strengths, a common drawback is the need for pipettes, syringes, ampoules or tubing for manipulating the liquid cesium or rubidium. This limits cell filling to a sequential rather than parallel process, or introduces technical difficulties in microfluidic manipulation of volatile alkali metals, and leads to variability from one cell to another.

To overcome these disadvantages and simplify the cell filling process, we have developed a cesium batch-filling technique based on thin film deposition and subsequent decomposition of cesium azide (CsN_3). CsN_3 is a solid at room temperature, is stable in air, and decomposes to produce cesium and nitrogen gas when heated to 450 °C in air or, as we have found, when exposed to UV radiation at room temperature. This method of cell filling eliminates the need for microfluidic manipulation of pure alkali metals since the cesium is fabricated inside the cell in-situ after the cell is already hermetically sealed, thus simplifying the filling process and reducing tooling costs. Furthermore, this method, being based on thin film deposition, is inherently wafer-level and lends itself to better uniformity across samples. However, due to the relative lack of thermophysical data on CsN_3 , as well as the lack of published literature concerning the material properties of CsN_3 , much of this work was exploratory in nature. While the photolysis of some other metal azides such as barium azide [9] is well known, the photolysis of CsN_3 has not, to our knowledge, been previously

demonstrated or studied, and as such the mechanisms for the photodissociation are not well understood.

EXPERIMENTAL METHODS

As described in previous work [4], a typical microfabricated alkali vapor cell is a silicon chip with a hole in it, sealed on each side by anodically bonded glass. The cavity usually contains buffer gases such as nitrogen, argon, or neon to preserve the coherence between the hyperfine components of the atomic ground state by preventing wall collisions. Figure 1 shows our process for fabricating, filling and sealing in parallel an array of cells on a wafer. The starting material is a 1 mm-thick, double-sided polished <100> silicon wafer. Cavities of dimension 1 mm x 1 mm were simultaneously etched through the wafer from both sides in KOH solution. Once the etch had penetrated through the wafer, thus opening up the <110> planes, the cavities were over-etched to produce straighter sidewalls. Deep reactive ion etching was also used to produce cavities with vertical sidewalls, but was found to be less efficient for these thick wafers because of problems in preserving the masking materials during the long etch.

After etching the array of through-holes, a 125 μm -thick piece of Pyrex was anodically bonded to one side of the silicon. The resulting silicon-glass stack is now referred to as a “preform.”

The next step is to deposit a thin film of CsN_3 into the array of cavities. Since CsN_3 is highly hygroscopic, a custom vacuum evaporator was constructed that was coupled to a nitrogen glove box such that the deposited films could be stored under inert conditions after deposition. Solid crystals of CsN_3 , obtained commercially, were ground into a fine powder and placed in an alumina crucible heated by tungsten basket filaments. An aluminum foil shadow mask was placed over the preform cavities. After the chamber was pumped to a base pressure of 1.33×10^{-4} Pa (1×10^{-6} Torr), the CsN_3 was heated in stages at a rate of $2^\circ\text{C}/\text{min}$, held at 250°C for several hours, heated to the melting point of 310°C , and held at this temperature for several minutes.

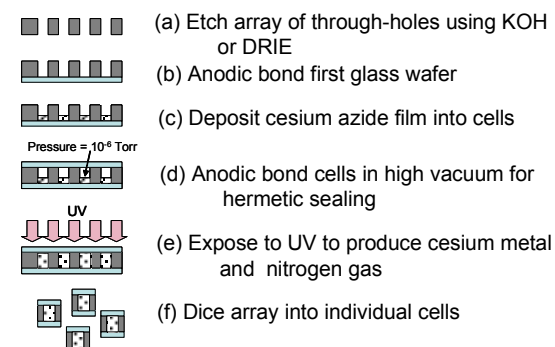


Figure 1. Cesium cell fabrication and filling process

The temperature was then increased to $325 - 360^\circ\text{C}$ and the shutter opened once a stable temperature and pressure were reached. Deposition rates varied from 0.1 to a few nanometers per second, with a pressure of 1.33×10^{-3} Pa (1×10^{-5} Torr). Typical thicknesses deposited were between 100 to 500 nm as measured by the crystal sensor, although, as discussed below, the occurrence of splattering and other instabilities makes the measured thickness somewhat variable.

Due to the relative lack of thermophysical data on CsN_3 , and also because CsN_3 is an ionic compound, the deposition process is slow, unstable, and not well understood. In general, direct thermal evaporation of compounds is difficult (or impossible, depending on the system in question) because of the tendency of the compound to break down into the constituent species due to the different vapor pressure of each species. Previous researchers have found that dissociation of CsN_3 in vacuum was negligible for temperatures up to 400°C [10]; nevertheless, we found a strong tendency for CsN_3 self-heating to occur, leading to rapid decomposition or explosion within the vacuum chamber. Thus, once melted, the CsN_3 is unstable and the exact nature of the deposition process is unknown due to simultaneous competing thermodynamic processes. We have observed that deposition of these films occurs through a combination of slow controlled vacuum evaporation and uncontrolled “splattering” of the evaporant, leading to highly nonuniform films that contain, at least partially, CsN_3 . Various combinations of thermal cycling, soaks at intermediate temperatures for various lengths of time, and adjusting the heating/cooling rates were found to stabilize the deposition process and reduce the likelihood of sudden or large fluctuations in temperature and pressure, to yield functional films.

The deposited films are nonuniform and consist of big agglomerates that range in size from a few micrometers to a hundred micrometers in diameter, as shown in Figure 2a. Energy dispersive spectroscopy indicated the presence of nitrogen in the films, as expected. Oxygen was also detected but its presence is inconclusive, possibly indicating a partial decomposition of the evaporant during deposition and producing cesium oxide, or else the oxygen detected could be that present in the aluminum substrate on which the films were deposited for EDS analysis.

Following CsN_3 deposition, the preform was placed in a custom vacuum anodic bonding chamber, baked overnight under vacuum, and then subsequently anodically bonded to another Pyrex piece at a temperature of 300°C , voltage of 1000 V, and chamber pressure of 2.6×10^{-4} Pa (2×10^{-6} Torr), thus enclosing a high vacuum inside the cell cavities. The sealed cell array was then exposed under a UV lamp of wavelength 254 nm and intensity of $2.3 \text{ mW}/\text{cm}^2$, over various lengths of time ranging from 8 hours to over 100 hours (depending on the thickness and nature of the

deposited film). Finally, the array was diced into individual cells. The cesium produced in-situ by the decomposition is visible under a microscope as gold-colored metallic droplets on the inner glass window (see Figure 2b). The N_2 gas produced from the reaction serves as a buffer gas to prevent collisions between the atoms and cell walls, and its pressure is proportional to the amount of azide dissociated. Because the residual azide is a thin film, the cell windows are relatively clean and optically transparent, more so than in our previous work in which the presence of bulk oxides or salts partially attenuated the laser light during cell testing or device operation.

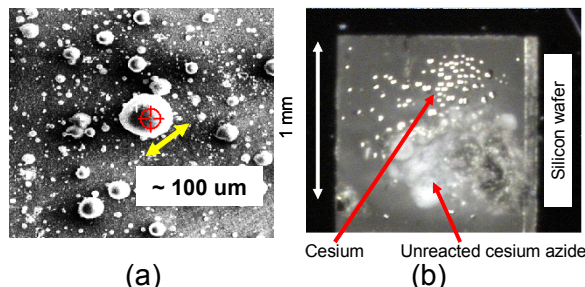


Figure 2. (a) Scanning electron micrograph of CsN_3 film. The cross indicates where EDS measurement was done. (b) Photograph of a cell as viewed through the glass window, showing in-situ production of cesium.

RESULTS

The photodecomposition of the CsN_3 into pure cesium and nitrogen gas was determined by cesium optical spectroscopy. The cell was heated to a temperature of $75 - 120$ °C to increase the cesium atomic density while the wavelength of a commercial vertical cavity surface emitting laser (VCSEL) was scanned over the cesium D2 transition at 852 nm by modulating the current with a triangular waveform. The resultant light then passed through the cell, and the two characteristic cesium absorption peaks, separated by 9.2 GHz corresponding to the cesium ground state hyperfine splitting, was detected by a commercial photodiode placed behind the cell. The broadening and shift of the spectral line with respect to a cesium cell without buffer gas may be used to estimate the pressure of the N_2 . Figure 3 shows typical absorption spectra from two different cells, containing different N_2 buffer gas pressures. Cells tested approximately 6 months apart show no change in the linewidth or absorption contrast.

Figure 4 shows a coherent population trapping resonance obtained from a CsN_3 -derived cell, although in this particular case the CsN_3 was not a deposited film but solid powder straight from the bottle and placed inside the cell cavity. The N_2 buffer gas pressure for this cell was about 37.3 kPa (280 Torr). Figure 4 shows that narrow linewidths are possible from CsN_3 -derived cells.

Another advantage of using MEMS technology to fabricate atomic vapor cells is the possibility of integrating other components directly

into the cell. For example, cell heating is necessary to increase the cesium vapor pressure for optical interrogation. Our approach to cell heating has been to sandwich the cell between heater chips, such heater chips being glass wafers on which indium-tin oxide films were deposited and used as thin-film heaters. Now, we have also taken advantage of MEMS technology to fabricate silicon heaters directly inside the cell (see Figure 5). The heater is designed as a bifilar winding to minimize the magnetic fields induced from the heating current.

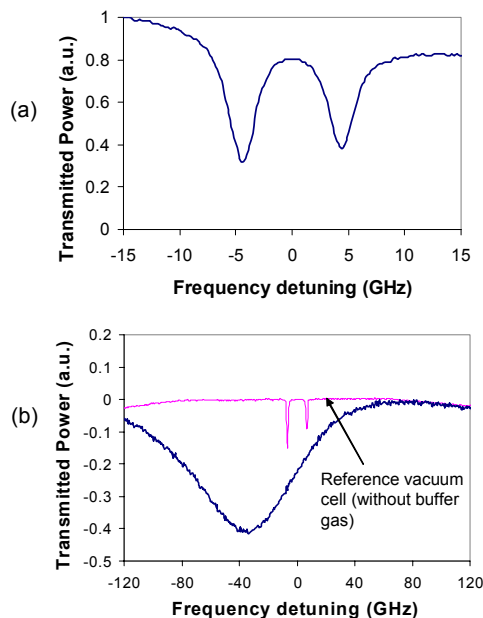


Figure 3. (a) Optical absorption spectrum of a cell with buffer gas pressure of about 26.7 kPa (200 Torr). (b) Optical absorption of a cell with high buffer gas pressure of about 133.3 kPa (1000 Torr, lower trace), which caused the two peaks to broaden until they are indistinguishable.

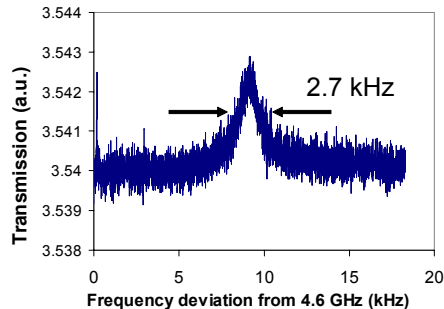


Figure 4. Cesium coherent population trapping resonance measured in a cell derived from UV photolysis of CsN_3 . The VCSEL was modulated with a RF frequency which was scanned in 20 kHz intervals around 4.6 GHz.

The winding is etched out of the silicon wafer by deep reactive ion etching, in the same process that defines the cell cavity, and boron doped to increase electrical conductivity. The heat conducts through the thickness of the silicon coil and thus heats both cell windows simultaneously. The boron-doped electrical traces on the silicon surface connect the heater inside the cavity to

contact pads at the edge of the cell while leaving the silicon surface topography unchanged for hermetic bonding to glass. Test cells of dimensions 1 cm x 1.1 cm x 0.35 mm were fabricated in this way, and were filled with cesium and about 26.7 kPa of N₂ and wirebonded to a metal chip carrier for table-top testing, as shown in Figure 5. The heater resistance was 350 ohms and 1 W of power was passed through the silicon heater. This power heated the cell as well as the metal chip carrier (see Figure 6) during the cesium spectroscopy such that the cesium vapor pressure was increased enough to provide the absorption peaks. The high power consumption was due to the fact that the test cell was much larger than that used in CSACs and the metal chip carrier acted as a heat sink.

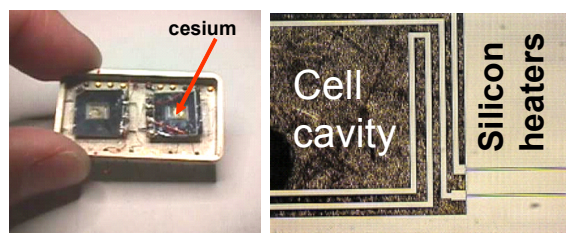


Figure 5. (a) Photograph of two test cells containing in-situ heaters, wirebonded to a metal chip carrier. (b) Photograph close up of cell heater.

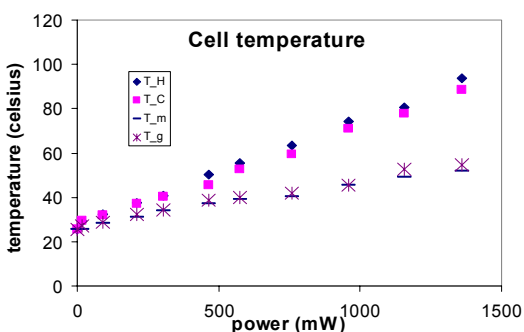


Figure 6. Temperature of various parts of the cell and chip carrier as a function of heater power. T_H = temperature of the glass window above the heating coil; T_C = temperature of the glass window above the center of the cell; T_m = temperature of the outersurface of the metal chip carrier; T_g = temperature of the glass thermal isolation spacer inserted between the cell and chip carrier.

Summary

We described a wafer-level technique for filling microfabricated resonance cells with cesium and nitrogen buffer gas for chip-scale atomic devices. A thin film of cesium azide was deposited into a wafer of cells; the wafer of cells was then hermetically sealed in vacuum, and the film was decomposed by UV exposure to produce pure cesium and nitrogen gas in-situ. Cesium optical absorption and coherent population trapping resonances were obtained from these cells. This cell filling technique is inherently wafer-level

compatible since it is based on thin-film deposition, and eliminates the need for microfluidic handling of alkali metals. Furthermore, the cells are optically transparent, and the buffer gas pressure may, in principle, be precisely controlled through the decomposition reaction. We also described monolithically integrated silicon cell heaters that can be used to heat the cell internally.

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