

Atomic vapor cells for chip-scale atomic clocks with improved long-term frequency stability

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A novel technique for microfabricating alkali atom vapor cells is described in which alkali atoms are evaporated into a micromachined cell cavity through a glass nozzle. A cell of interior volume 1 mm^3 , containing ^{87}Rb and a buffer gas, was made in this way and integrated into an atomic clock based on coherent population trapping. A fractional frequency instability of 6×10^{-12} at 1000 s of integration was measured. The long-term drift of the $F=1$, $m_F=0 \rightarrow F=2$, $m_F=0$ hyperfine frequency of atoms in these cells is below $5 \times 10^{-11}/\text{day}$.

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The union of atomic spectroscopy and fabrication techniques used in microelectromechanical systems (MEMS) continues to open up new possibilities for the development of miniature atom-based instruments. Recently, the first physics packages for chip-scale atomic clocks^{1,2} and magnetometers³ have been demonstrated. The use of MEMS techniques not only allows for miniaturization and more favorable power consumption but also permits simpler and more robust designs. Furthermore, wafer-level fabrication and integration could substantially reduce fabrication costs. At the same time, scaling devices to smaller sizes does not necessarily degrade their performance substantially. Chip-scale atomic clocks (CSACs) with fractional frequency instabilities $\leq 6 \times 10^{-11}/\tau^{1/2}$ (Ref. 4) and chip-scale magnetometers with magnetic field sensitivities below $1 \text{ pT/Hz}^{1/2}$ (Ref. 3) seem possible. For the clocks this represents a degradation by only a factor of 5 from existing commercially available devices, while the volume and power consumption of the device would each potentially be reduced by nearly 2 orders of magnitude.

One of the biggest advantages of using atoms as the frequency-defining element rather than quartz-crystal resonators is the potential of much better frequency stability at long integration times. Until now, however, little attention has been paid to the long-term performance of microfabricated atomic clocks. The first chip-scale alkali vapor cells, which were made by anodic bonding of silicon and glass,^{5,6} showed substantial fractional frequency drifts of $\sim -2 \times 10^{-8}/\text{day}$ (see Fig. 1) as well as temperature coefficients of the fractional frequency near $+2.3 \times 10^{-8}/\text{K}$. Those first cells were filled with alkali atoms by reacting roughly $100 \mu\text{g}$ of barium azide with alkali chloride directly in the cell at elevated temperature. The reason for the observed frequency drift is the presence of impurities, namely, barium, chlorine, and some nitrogen, that remain in the cell after it is sealed. Since the cell dimensions are only $\sim 1 \text{ mm}$, the cells must be heated to between 85 and 95°C to reach the density of the alkali vapor necessary to produce optimal absorption (roughly 50%) of the resonant light. At these temperatures, nitrogen and barium can recombine to form barium azide.

This slowly and continuously depletes the nitrogen buffer gas pressure inside the cell. Since the clock frequency shifts with nitrogen pressure by roughly $+6.7 \times 10^{-7}/\text{kPa}$ for Cs and $+6.0 \times 10^{-7}/\text{kPa}$ for ^{87}Rb ,⁷ the decreasing N_2 pressure causes a time-varying frequency shift. The frequency shift rate measured for the Cs cell, for example, would correspond to a N_2 depletion rate of roughly 30 Pa/day . Furthermore, the amount of nitrogen trapped by the barium depends on the cell temperature, resulting in a temperature-dependent frequency shift much larger than expected for the combination of buffer gases used. In one previous attempt to reduce these two shifts,⁴ microfabricated cells were made with the same chemical reaction method but without intentionally introducing nitrogen to the cell. Before the cell was sealed, the nitrogen produced in the reaction was pumped away and the cell was instead filled with a combination of neon and argon. Inert gases do not react with the barium residue, and a reduced frequency drift was therefore expected. Indeed, a chip-scale clock physics package based on a cell made in

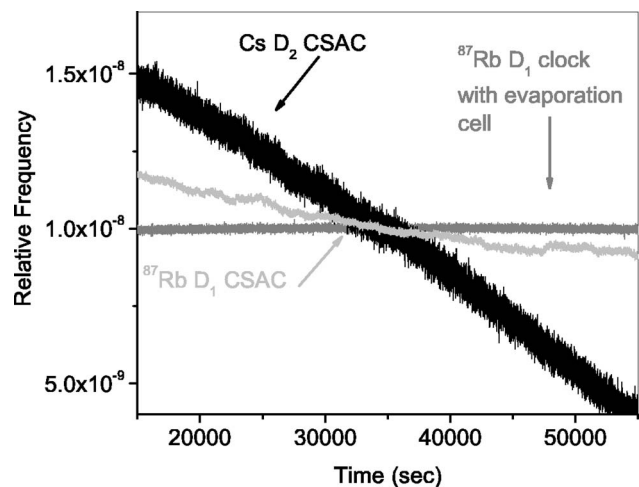


Fig. 1. Output frequency of the CPT clock, normalized to the frequency of the ground-state hyperfine splitting of the alkali atom, for Cs atoms excited on the D_2 line CSAC (black), ^{87}Rb atoms excited on the D_1 line CSAC (light gray) and the ^{87}Rb D_1 line frequency reference based on the cell fabricated with the new evaporative filling method (gray).

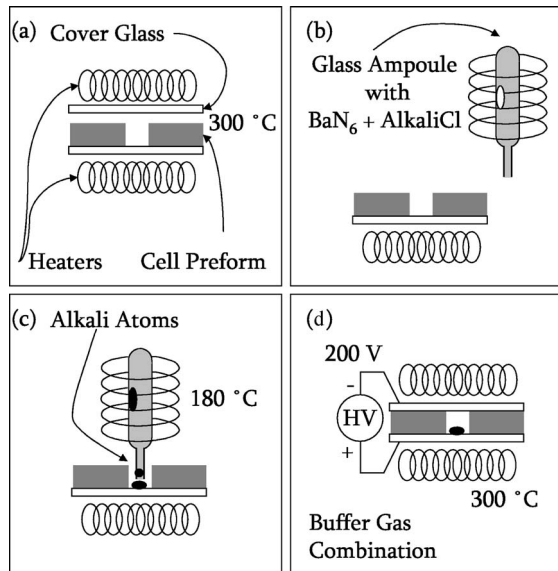


Fig. 2. Novel filling method for chip-scale alkali vapor cells. (a) The silicon and glass preform and the cover glass are baked at 300 °C inside the vacuum chamber. (b) A mixture of barium azide and alkali chloride is reacted inside a glass ampoule to create barium, chlorine, alkali atoms, and nitrogen; the nitrogen is pumped away. (c) The alkali atoms are diffused through the glass nozzle into the cell preform. (d) The chamber is then filled with an appropriate mixture of buffer gases. Finally, the cover glass is anodically bonded to the silicon preform at a temperature of 300 °C and with 200 V applied across the sample. HV, high voltage.

this way showed an improved frequency drift of -5×10^{-9} /day (Fig. 1). The residual drift was most likely caused by some smaller amount of nitrogen left inside the cell, either because it was not fully pumped off before the cell was sealed or because the reaction was not fully completed before the final bonding step was carried out.

To address these problems, we developed a new method for filling microfabricated vapor cells that leaves no substantial residue of chemicals inside the cell (Fig. 2) and thus results in much improved long-term frequency stability. A cell preform was made from a piece of silicon 1 mm thick through which a hole of cross-sectional area 1 mm² was etched by potassium hydroxide etching. The silicon wafer was anodically bonded to a 300 μm thick piece of borosilicate glass to create a cavity for the atoms of volume 1 mm³. Then the preform and top glass wafer were baked at a temperature of 300 °C for 12 h at an ambient pressure near 10⁻⁴ Pa [Fig. 2(a)].

The alkali atoms are produced by the chemical reaction of barium azide and alkali chloride inside a glass ampoule of 4 mm diameter and 3 cm length, which had been thinned out on one side to form a nozzle of roughly 1 cm length and diameter of 700 μm [Fig. 2(b)]. Using the barium azide and rubidium chloride approach rather than rubidium metal allows us to make cells with isotopically selected ⁸⁷Rb, which show better performance in a clock than cells with the natural mixture of rubidium isotopes. The chemical reaction was controlled by a heater around the ampoule, and the reaction rate

was monitored by measuring the nitrogen pressure with a mass spectrometer. As the ampoule was heated, a small ball of alkali atoms formed on the tip of the nozzle. The tip was then lowered into the cavity of the cell preform [Fig. 2(c)]. The alkali atoms were evaporated through the nozzle into this cavity. The second piece of borosilicate glass, of 300 μm thickness, was placed on top of the cell preform. After the nitrogen pressure inside the vacuum chamber dropped below 10⁻⁵ Pa, the vacuum chamber was filled with the mixture of neon and argon. To reduce the amount of water and oxygen in the buffer gases that could react with the alkalis deposited into the cell, the cover glass was pressed against the cell preform while the chamber was backfilled with the buffer gases. The barium and alkalis remaining inside the ampoule and some alkalis that had diffused onto a metal holder acted as getter, purifying the buffer gas by absorbing residual oxygen. After some time, the top cell window was removed again to allow the buffer gases to fully enter the cell. Finally, the cell was closed by anodic bonding of the top glass to the silicon preform at a temperature of 300 °C, and 250 V was applied across the sample [Fig. 2(d)].

Cells containing ⁸⁷Rb made with this method were characterized using a tabletop coherent population trapping (CPT) apparatus.⁸ A vertical-cavity surface-emitting laser (VCSEL) at 795 nm delivered light to the atoms via a micro-optics assembly, consisting of a spacer, a microlens, two neutral-density filters, and a quarter-wave plate. The light passed through the cell, which was mounted between two aluminum disks that were heated to 90 °C with heaters made from twisted wires. The cell was shielded from laboratory magnetic fields with a cylinder of high-permeability material, and a longitudinal magnetic field of 42 μT was applied to lift the degeneracy of the Zeeman ground states. The light transmitted through the cell was detected with a photodiode. The VCSEL injection current was modulated at 3.4 GHz, and roughly 60% of the light was transferred into the two first-order modulation sidebands. The modulation frequency was then locked to the CPT resonance coupling the magnetically insensitive (to first order, $m_F=0$) Zeeman components. This locked frequency was compared with a more stable frequency reference. The frequency drift, shown in Fig. 1, was reduced to below 5×10^{-11} /day. This number is limited by the accuracy of our measurement system, partly due to the presence of a frequency shift with temperature of $2.5(7) \times 10^{-9}$ /K. We believe that the residual contamination of the cell with barium is no longer a limitation to the frequency stability for integration times below 1 day.

This system achieved a fractional frequency instability, characterized by the Allan deviation, of $6 \times 10^{-11}/\tau^{1/2}$ (Fig. 3) at short integration times, averaging down to 6×10^{-12} at 1000 s of integration. At 1 h of integration, the fractional frequency instability was 8×10^{-12} . We believe that the remaining frequency instability at long integration times is caused by variations in the pointing direction of the laser beam due to thermomechanical motion of the optical

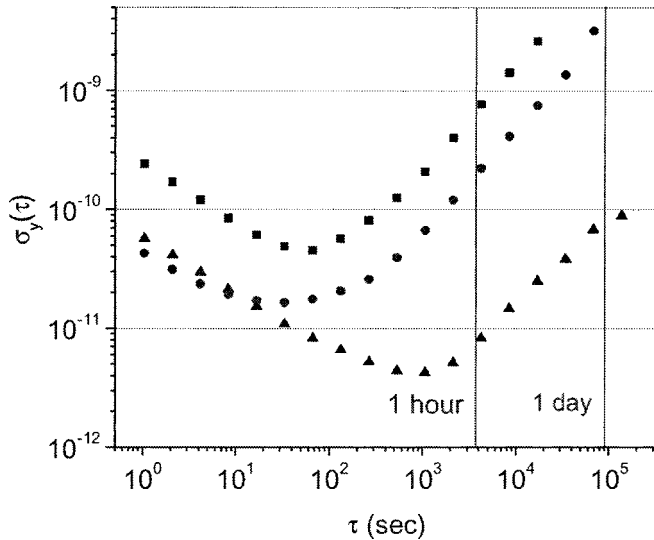


Fig. 3. Fractional frequency instability characterized by the Allan deviation $\sigma_y(\tau)$ as a function of averaging time of the Cs D_2 CSAC (squares) and the ^{87}Rb D_1 CSAC (circles) compared with the instability of the frequency reference based on a cell made with the new evaporative filling method (triangles). This last clock was based on the D_1 line of ^{87}Rb .

components used to direct the beam, which changes the AC Stark shift of the CPT resonance. This instability would be reduced if the cell were mounted in a compact, integrated physics package such as that described in Ref. 1.

In conclusion, we have described a novel method for filling chip-scale alkali vapor cells that shows significantly improved long-term frequency stability compared with cell-filling methods developed previously. The improved stability is due to reduced linear frequency drifts and smaller temperature-related effects. The measurements presented here demonstrate that it is possible to achieve long-term instabilities $\leq 10^{-11}$ at 1 h of integration in microfabricated cells. This suggests that chip-scale

atomic clocks will be a viable technology for applications that require small size, low power consumption, and long-term frequency stability.

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