Microfabrication of ^{85}Rb Vapor Cell for Chip-Scale Atomic Clocks

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***Abstract*—We describe the fabrication of Rubidium (Rb) vapor cells using glass-silicon anodic bonding techniques and chemical reaction between rubidium chloride and barium azide. The pure metallic Rb drops and buffer gases were obtained in the cells with few mm^3 internal volumes. The optical absorption resonance of ^{85}Rb D1 transition with proper broadening and the corresponding coherent population trapping (CPT) resonance, with a signal contrast of 1.5% and linewidth of about 1.7 KHz, have been detected at 90℃.**

I. INTRODUCTION

In recent years, chip-scale atomic clocks (CSACs) have been increasingly recognized to be significant in the man-portable devices such as the synchronization of telecommunications networks, global positioning system (GPS) receivers, and many measuring instruments for their requirements of ultra-low power consumption, small size, and low fabricating cost. The direction to miniaturize the size and power consumption of microwave atomic clocks is achieved by factors of low power vertical cavity surface emitting laser (VCSEL), coherent population trapping (CPT) technique, and small-size alkali vapor cells. In traditional microwave atomic clocks, the size of vapor cells has been limited by the wavelength of the excitation microwave. This limitation can be cancelled by introducing the CPT technique into the realization of atomic clocks[1-2]. In CPT based atomic clocks, when the frequency difference of the two coaxial laser beams is equal to the frequency separation of the hyperfine split ground state of the atoms, the atoms are optically pumped into CPT state, a reduction of the optical absorption could be observed. Such a working principle allows the vapor cells to be reduced to sub-millimeter scale. Therefore, the microfabrication of vapor cells has proven a challenging task. Thus, a microfabrication process of the atomic vapor cell by using the techniques of micro-electromechanical systems (MEMS) has been proposed to create a "glass-silicon-glass" sandwich structures with alkali atoms (cesium (Cs), or rubidium (Rb)) and buffer gases sealed in the hole of the silicon plate by using anodic bonding technique[3]. Considering the conflict between high temperature requirements during the anodic bonding process and the low melting points of Cs or Rb, to fill alkali atoms into the holes has been proven to be another technique challenge. There are several cell-filling techniques to introduce Cs or Rb into vapor cells being explored for this kind of vapor cell microfabrication such as direct injection of the alkali metal liquid, in situ chemical or electrochemical reactions to produce alkali metals during or after the sealing process[3-7].

Typically, 87Rb and Cs have been used in CPT based atomic clocks. For Cs, its vapor pressure is higher than Rb's at the same temperature which can lower the operating temperature of the cells in atomic clocks. However, as indicated by R. Lutwak et al, due to Cs' s higher Zeeman degeneracy compared with Rb' s, the CPT resonance contrast for both D1 and D2 excitation in Cs are lower than that in Rb [8]. Moreover, it has been proven that the CPT signal strength excited on the D1 transition is much higher than that on D2 transition in Rb theoretically and experimentally, but such advantage is less pronounced in Cs[9]. Therefore, microfabrication of Rb cells is also of significance in CSACs' development. Recent years, many groups developed various methods to fabricate Rb vapor cells[10-12]. We choose ^{85}Rb rather than traditional ^{87}Rb for its lower ground state hyperfine splitting frequency, which can lower the required power of the laser modulation sources and is more effective for the circuit designs of radio frequency (RF)[13]. In this paper, we fabricated ^{85}Rb vapor cells based on MEMS technology. By using the similar method employed by S. Knappe et al[12] and controlling the pre-treatment and reaction conditions, we achieved appropriate buffer gas pressure of nitrogen (N_2) in sealed 1mm long cells so that we obtained clear CPT signals of ^{85}Rb D1 line with linewidth of about 1.7kHz at 90℃.

II. EXPERIMENTAL METHOD

The experimental procedure is schematically shown in Fig. 1. The silicon plate was double polished with a thickness from 0.5mm to 1 mm, and was p-doped with <100> crystal direction. A through hole with diameter ranging from several hundred microns to a few millimeters was elaborated in the silicon plate by laser ablation or ultrasonically drilling. A Pyrex glass plate with 500 microns thick was photolithographically patterned as the same size of above mentioned silicon plate. Then the two plates were anodically bonded together as shown in Fig. 1(a).

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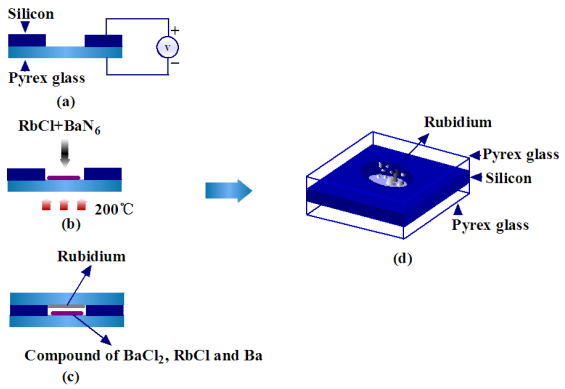

Fig. 1 Schematic illustration of the Rb vapor cell fabrication procedures. (a) Holed-silicon plate was anodically bonded to a Pyrex glass. (b) The compound of RbCl and BaN_6 was put into the hole and heated at 200℃ to release N_2. (c)The second Pyrex glass plate was bonded to complete the sealing. (d) A completed cell with metal drops in the inside surface of one Pyrex glass plate.

Then, we put the mixture of rubidium chloride (RbCl) and barium azide (BaN_6) into the hole to get pure Rb metal as well as N_2 buffer gas (Fig. 1(b)). The process of the reaction between these two chemicals can be summarized as follows:

$$BaN_6 \xrightarrow{200°C} Ba + 3N_2 \uparrow \quad (1)$$

$$2RbCl + Ba \xrightarrow{250-300°C} 2Rb \uparrow + BaCl_2 \quad (2)$$

These chemical reactions are firstly employed by S. Knappe et al[12], where they used ^{87}RbCl as the reactant and consequently obtained ^{87}Rb. Here we used the common chemical RbCl (99% purity, Alfa Aesar) to generate natural mixture of Rb isotopes to demonstrate the feasibility of our experimental procedure. The mixture is composed of RbCl and BaN_6 powder with the stoichiometric ratio according to the above reaction equations (the mass ratio of RbCl to BaN_6 is 1:0.92), and actualized by mixing them together through grinding, or dissolving them into water to form a clear colorless solution which was then baked to produce white solid powder.

After putting the compound into the hole, we placed the plate on the hot plate in the high vacuum chamber and connected it to electrodes. The pressure of the chamber can reach 4.5×10^{-4}Pa which is low enough to avoid the reaction of Rb with some elements in the air. The modulatory ranges of the hot plate temperature and the voltage between electrodes are large enough to ensure the anodic bonding. At first, the hot plate was heated to 200℃ and kept for a few hours to make sure that BaN_6 was decomposed into N_2 and Ba completely, and the change of the pressure in the chamber was monitored by vacuum gauge to ensure there was enough N_2 being left as buffer gas in the mean time. Then, the open side of the hole is covered with the second Pyrex glass, and the temperature of the hot plate was increased to 250℃ and 1000V voltage was applied to initiate the anodic bonding in the contacted surface to seal the hole hermetically. In the mean time, pure Rb vapor was generated through the reaction of RbCl and Ba. Subsequently, we left the sealed assembly with a sandwich structure in the chamber at around 300℃ for at least 30 minutes to guarantee the compound was reacted completely (Fig. 1(c)). Finally, a three-layered Rb vapor cell was obtained with metal drops coagulating on the internal surface of one Pyrex glass (Fig. 1(d)).

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. Observation of the Rb in the Cell

Fig. 2 shows the photograph of the successfully sealed Rb vapor cell, in which the shiny drops on the inside surface of one Pyrex plate is the metallic Rb, and the black solid materials besides the Rb drops are the compound of Ba, $BaCl_2$, and the minimum unreacted RbCl. In Fig. 2, the diameter of the hole was 3 mm to show the rubidium metal drops more clearly.

Fig. 2 Photograph of a sealed cell (10×20×1㎜). Shiny Rb drops are visible.

B. Optical Absorption Resonance

The existence of Rb and N_2 produced from chemical reaction in the cell was confirmed by optical absorption spectroscopy. Fig. 3 shows the schematic illustration of experimental setup for measuring the optical absorption resonance and CPT resonance frequency in ^{85}Rb vapor cells. By modulating the sweeping current, the wavelength of the commercial VCSEL was scanned over the ^{85}Rb D1 transition at 795 nm. Then the laser beam passed through the vapor cell, which was heated to a controlled temperature ranging from room temperature to 120℃, and the transmitted light power was recorded using a photodiode and shown on a oscillograph at the same time.

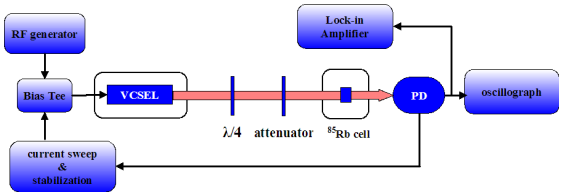

Fig. 3 Schematic illustration of experimental setup for measuring the optical absorption resonance and CPT resonance frequency

As shown in Fig. 4(a), at 80℃, the two absorption resonance of ^{85}Rb separated by the ground state hyperfine splitting of ^{85}Rb (3.04GHz) began to be observed. The absorption became obvious at 90℃. The width of one of the absorption resonance is about 1 GHz at 90℃. We could evaluate the pressure of the buffer gas by comparing the

broadening of the absorption line with the cell made without buffer gas[14]. By this method, we estimated the buffer gas pressure in it is below 10^2 kPa. In contrast, we also obtained some absorption curves with broaden line of widths of about 10GHz or larger at different temperatures in other vapor cells, in which the buffer gas pressure exceeded 10^2 kPa, shown in Fig. 4(b). This very large broadening is caused by the excessive collisions between Rb atoms and buffer gas molecules. With such amount of buffer gas in vapor cells, we cannot lock the laser wavelength on the right transition that CPT signal is hard to be detected.

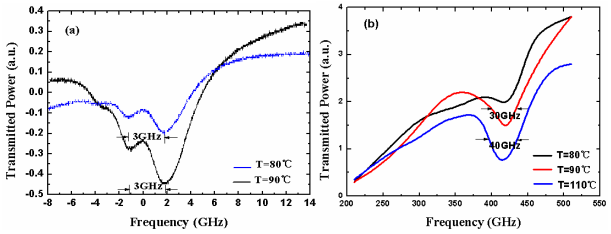

Fig. 4 Characteristic optical absorption spectra of ^{85}Rb vapor sells at different temperatures. (a) Two lines under buffer gas pressure below 10^2 kPa; (b) broaden overlapped peak under buffer gas pressure exceeded 10^2 kPa.

C. CPT Resonance

Fig. 5 shows the CPT resonance in the cell. In this experiment, the VCSEL beam (2 mm in diameter) is circularly polarized and attenuated that only 100μW left before entering the vapor cell. With the modulation of sweeping current, a substantial fraction of the laser power is transferred to the sidebands from the carrier band. By locking the VCSEL beam frequency on the absorption spectrum, the first sideband is roughly matched with the transition to create the resonance. Shown in Fig. 5(a), the observed linewidth of the CPT resonance is about 1.7 kHz, and the contrast is about 1.5%. Fig. 5(b) shows its related error signal. This narrow linewidth of the measured CPT resonance suggests that the cell made from the chemical reaction is possible to be used in CSACs and other devices based on the same principle.

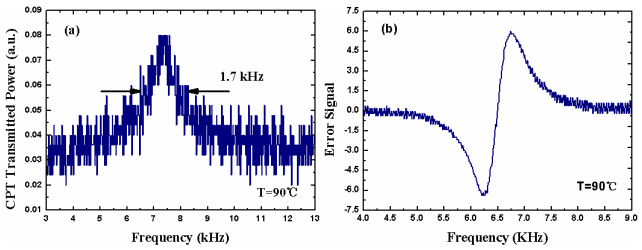

Fig. 5 Measured CPT resonance signal of the cell at 90℃ has width of 1.7 kHz and signal contrast of 1.5%; (b) Related error signal.

IV. CONCLUSIONS

By using the chemical reaction between RbCl and BaN_6 to produce pure Rb metal in atomic vapor cells, we fabricated the ^{85}Rb vapor cells successfully. The optical absorption resonance of ^{85}Rb was detected in the sealed cells. We also got the CPT resonance signal with contrast of 1.5% and width of about 1.7 KHz, which suggests that this kind of cells can be applied in miniaturized atomic clocks based on CPT resonance. Furthermore, by optimizing the detective methods and vacuum condition in the chamber, we could control the buffer gas pressure more precisely so that its use can be widen to other frequency instruments based on atomic excitation.

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