Influence of architecture and temperature of alkali atom vapor cells on absorption spectra

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Abstract: Chip-sized alkali atom vapor cells with high hermeticity are successfully fabricated through deep silicon etching and two anodic bonding processes. A self-built absorption spectrum testing system is used to test the absorption spectra of the rubidium atoms in alkali atom vapor cells. The influence of silicon cavity size, filling amount of rubidium atoms and temperature on the absorption spectra of rubidium atom vapor in the atom vapor cells are studied in depth through a theoretical analysis. This study provides a reference for the design and preparation of high quality chip-sized atom vapor cells.

Key words: chip-sized alkali atom vapor cell; absorption spectrum; silicon cavity; rubidium azide

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1. Introduction

Many physical studies and precise measurements are based on the quantum process of the interaction between light and atoms. To prepare long-lived atomic polarization state in atomic vapor, it is necessary to break the atomic energy level population formed by the Boltzmann distribution with the help of an external influence. There are many kinds of quantum sensing instruments based on chip-sized atom vapor cells, such as atomic clocks^[1-3], atomic magnetometers^[4–5], atomic gyroscopes^[6–7], and so on. These instruments utilize the discontinuous and guantized atomic energy level structure of the alkali atoms in the atom vapor cells to achieve their measurements. However, the discontinuity of the atomic energy level makes frequency, wavelength and other physical quantities related to the energy level no longer continuous. The physical quantities such as frequency and wavelength have a definite quantization state, and thus have extremely high precision and stability. Therefore, the interaction process between a high-performance chip-sized atom vapor cell and light is necessary for high-precision measurements of quantum sensing instruments, which lays a good foundation for the realization of chip based measurements and sensing tests.

There has been much research on the alkali atom vapor cells. Tsvetkov's group^[8] achieved a high density alkali atom vapor in a polydimethylsiloxane coated cell at room temperature by studying LIAD (light induced atomic desorption), which can achieve higher by two orders of magnitude than the thermodynamic equilibrium value. Kiyose's group^[9] fabricated the atom vapor cell containing a pipetting cavity, an optical cavity, and a microchannel connecting the two cavities by using MEMS technology. The stability of the Cs atomic density in the atom vapor cell was confirmed by measuring the

Correspondence to: W Li, livy09@163.com Received 8 JULY 2022; Revised 4 AUGUST 2022. ©2022 Chinese Institute of Electronics absorption spectrum of cesium atoms over a period of 5 months. Hasegawa's group^[3] prepared the caesium atom vapor cells filled with Ar or Ne buffer gas by using two-step anodic bonding technology, and got the appropriate pressure of the atom vapor cells filled with buffer gases that can meet the needs of atomic clocks by studying spectroscopy. NIM (National Institute of Metrology, China)^[10] reported a millimeter-level alkali atom vapor cell with a high hermeticity for chip scale atomic clocks, whose leakage rate was up to 5 \times 10⁻⁸ mbar·L/s. Two sealed cavities were prepared through a wet etching and single-chip anodic bonding process. The absorption spectrum and coherent population trapping (CPT) resonance linewidths were reduced by optimizing the buffer gas pressure in vapor cells. Researchers have been making great efforts to improve the stability of the vapor cells and increase the atomic life in the vapor cells by studying the spectroscopy of alkali atoms. However, the influence of silicon cavity size, filling amount of alkali atoms, and temperature on the absorption spectra of alkali atoms in the atom vapor cells is rarely reported, which is of great significance for the design and fabrication of chip-sized atom vapor cells.

In this work, alkali atom vapor cells with different silicon cavity sizes and filling amounts of rubidium atoms are prepared by micro-electromechanical system (MEMS) technology. The high hermeticity atom vapor cells are successfully fabricated by deep silicon etching and two anodic bonding processes. A self-built absorption spectrum testing system is used to test the absorption spectra of rubidium atoms in the atom vapor cells with different silicon cavity sizes, different filling amounts of rubidium atoms, and different temperatures, which provides a reference for the design and preparation of high quality chip-sized alkali atom vapor cells.

2. Experiments

2.1. Preparation of chip-sized alkali atom vapor cells

The chip-sized alkali atom vapor cell, as shown in Fig. 1, has a glass/silicon/glass structure. There is a silicon cavity in



Fig. 1. Image of chip-sized alkali atom vapor cells.



Fig. 2. (Color online) The preparation process of MEMS atom vapor cell.

the middle of the atom vapor cell, which is used to store rubidium atom vapor and buffer gas and provide a place for the interaction between light and alkali atoms.

In this work, a 4-inch diameter and 1 mm thick N-type <100> silicon wafer that is polished on both sides is chosen. A 4-inch borosilicate glass wafer with low surface roughness and a thermal expansion coefficient similar to that of the silicon wafer is selected for bonding to the silicon wafer by anodic bonding method. Rubidium azide (RbN₃), which is chemically stable at room temperature is filled in an atom vapor cell and is then thermally decomposed into rubidium atoms and nitrogen under laser irradiation. The photolysis method is simple, easy to implement, and free of impurities^[11].

Chip-sized alkali vapor cells are prepared by using deep silicon etching technology, anodic bonding technology, and the photolysis method. The preparation process of MEMS alkali vapor cell is shown in Fig. 2. First, a 1 μ m thick aluminum film is grown on the cleaned silicon wafer as a hard mask layer by using a magnetron sputtering machine. The silicon wafer is processed by coating photoresist, pre-baking, exposing, developing and post-baking. It is then put into a mixed acid etching solution (mainly phosphoric acid) to etch the aluminum film. After the pattern on the aluminum film is formed, the silicon wafer is cleaned and dried. A 500 nm aluminum film is then grown on the backside of the silicon wafer using the magnetron sputtering machine as a protective layer for the substrate of the etching machine. Throughcavities are etched with a plasma etching machine. The mixed acid etching solution is used to remove aluminum films on both sides of the silicon wafer. Then, silicon cavities are obtained. The first anodic bonding is performed on the glass wafer and the silicon wafer with cavities. After bonding, a layer of silver glue is evenly spreaded on the side of the silicon wafer/glass wafer in a segmented manner, and then is dried. Quantitative RbN₃ solution is filled into the silicon cavity with a microsyringe and the moisture is dried with a hot plate. Next, the second anodic bonding is carried out to form a glass wafer/silicon wafer/glass wafer three-layer sealing structure which is then divided into small units of $1 \times 1 \text{ cm}^2$ with a grinding wheel scribing machine. The small units are then irradiated with a deuterium lamp for 3 h to decompose RbN₃ into Rb and N₂. Finally, the alkali atom vapor cells are successfully achieved.

In this work, chip-sized alkali atom vapor cells with different silicon cavity sizes are prepared. The diameters of the silicon cavities are 4, 5, and 6 mm, respectively. In addition, the silicon cavities are filled with RbN₃ with a mass of 250 μ g. Another batch of alkali atom vapor cells are prepared with different filling amounts of RbN₃, which are 125, 250, 375, and 500 μ g, respectively. The silicon cavities are all 4 mm in diameter.

2.2. Tests of chip-sized alkali atom vapor cells

The hermeticity of alkali atom vapor cell is characterized by the leakage rate. According to GJB 548B-2005^[12], the test method of the leakage rate is as follows. Because the maximum volume of the silicon cavities prepared in this work is 36 mm³ (<0.05 cm³), the alkali atom vapor cells are placed in a sealed chamber and pressurized to 517 kPa with helium for 2 h. After they are taken out from the sealed chamber, the alkali atom vapor cells are degassed by N₂ purge to remove the helium adhered to the surface of the vapor cells. Then, the leakage rate of the vapor cell is measured in a helium mass spectrometer within 1 h. After testing, the average leakage rate of the alkali atom vapor cells with different silicon cavity sizes prepared in the same batch is $1 \times$ 10^{-9} Pa·m³/s, while the average leakage rate of the vapor cells prepare in the same batch with different contents of RbN₃ is 1.3×10^{-8} Pa·m³/s. It has been reported^[13] that the leakage rate of the alkali atom vapor cell prepared by the low temperature anodic bonding method is 2.8×10^{-7} Pa·m³/s, which confirms that the hermeticity of alkali atom vapor cells in this work is in comparison much better. The difference in the leakage rates of the alkali atom vapor cells between the two batches is mainly caused by the different preparation processes.

An absorption spectrum testing system is built in this work. A schematic diagram of the system is shown in Fig. 3, which mainly includes a light source, light source collimating and focusing lenses, a sample, a monochromator, a detector, a lock-in amplifier, and a computer. The light source has broad band wavelength which covers the wavelength of the alkali atomic absorption line spectrum. The chopper modulates the light beam intensity and provides the reference frequency for the lock-in amplifier. The light is focused on the sample through the collimating and focusing lenses. Atoms in the sample absorb photons, and then the transmitted light



Fig. 3. (Color online) Schematic diagram of the absorption spectrum testing system. Optical beams are shown in red lines. Electrical connections are shown in blue lines. Light source; C, chopper; L1, collimating lens; L2, focusing lens; S, Sample; L3, focusing lens; A1, entrance slit; Monochromator; A2, exit slit; PD, detector; Lock-in amplifier; Computer.



Fig. 4. (Color online) Energy level spectrum of the Rb atom.

is focused to the entrance slit of the monochromator by the focusing lens. The detector converts the optical signal which gets out from the exit slit of monochromator into an electrical signal. The electrical signal is input to the lock-in amplifier to improve the signal-to-noise ratio. Finally, the electrical signal connects to the computer to get the spectrum.

The energy level spectrum of the Rb atom is shown in Fig. 4^[14]. By optical fields at 795 and 780 nm, the S_{1/2} ground state is coupled to the two lowest-energy excited states $P_{1/2}$ and $P_{3/2}$, corresponding to the D1 and D2 lines, respectively. These transitions are usually excited by a lamp or a laser. Atoms or molecules in the ground state and low excited state absorb light with a continuous distribution of certain wavelengths and transition to each excited state, forming dark lines or dark bands arranged by wavelength in the absorption spectrum^[15]. Therefore, the filling of alkali atoms in the atom vapor cells can be characterized by the absorption spectrum of Rb atoms. The absorption spectra of alkali atoms in vapor cells with different silicon cavity sizes and different alkali atom filling amounts are measured to study the atomic densities of rubidium vapor in atom vapor cells.

3. Results and discussion

3.1. Absorption spectra of alkali atom vapor cells at different temperatures

The existence of rubidium atomic vapor in the chip-sized alkali atom vapor cell is verified by the absorption of rubidium atoms at specific wavelengths. The atom vapor cell containing rubidium atoms and nitrogen is heated to ensure a sufficient atomic density in the atom vapor cell. The absorption spectra of alkali atom vapor cells with the silicon cavity diameters of 4, 5, and 6 mm at different temperatures are measured by using the absorption spectrum testing system, as shown in Fig. 5. It can be seen that although the atom vapor cells are heated to 80 °C, the intensities of the absorption peaks are still relatively weak, which is due to the fact that the chipsized alkali atom vapor cells have only a 1 mm optical path. Two absorption peaks of rubidium atoms at 780 and 795 nm can be clearly seen from the absorption spectra. According to Fig. 4, these two absorption peaks correspond to the D2 line $(5\ ^2P_{3/2} \rightarrow 5\ ^2S_{1/2})$ and D1 line $(5\ ^2P_{1/2} \rightarrow 5\ ^2S_{1/2})$ of rubidium atom, which proves the existence of rubidium vapor in the atom vapor cells. In addition, with the increase of the temperature of the atom vapor cells, the full width at half maximum of the two absorption peaks of the rubidium atoms gradually increase, which is caused by the Doppler broadening effect.

For each alkali atom vapor cell heated from 80 to 140 °C, the absorption peak intensities at 780 and 795 nm become stronger with increasing temperature. The transmitted light intensity through the atom vapor cell can be expressed as the following formula^[16]

$$I_{\rm T} = I_0 \exp[-n\sigma(v)L],\tag{1}$$

where I_0 is the intensity of incident light, *n* is the atomic density of alkali atoms in the atom vapor cell, *L* is the length of the atom vapor cell, and $\sigma(v)$ is the photon absorption cross section, which is obtained from the following formula

$$\int_{0}^{+\infty} \sigma(v) dv = \pi r_{\rm e} c f_{\rm res}, \qquad (2)$$

where $r_{\rm e}$ is the electron radius, c is the speed of light in vacuum, and $f_{\rm res}$ is the transition oscillator strength, which corresponds to the proportion of a given resonance in the total cross section. For alkali atoms, the oscillator strengths are approximately given as $f_{\rm D_1} \approx 1/3$ and $f_{\rm D_2} \approx 2/3^{[17]}$.

As the temperature of the alkali atom vapor cell increases, the atomic density of the rubidium atoms in the vapor cell increases. It can be obtained from Eq. (1) that the intensity of the transmitted light through the atom vapor cell weakens. Namely the intensity of the light absorbed by the rubidium atoms in the atom vapor cell increases. So the intensities of the two absorption peaks in the absorption spectrum increase continuously with the increase of temperature. In addition, it can be seen from Fig. 5 that the absorption peak at 780 nm (D2 line) is stronger than that at 795 nm (D1 line) in each atom vapor cell. Because the transition oscillator strength of the D2 line of alkali atoms is stronger than that of the D1 line. It is obtained from the Eq. (2) that the transition photon absorption cross section of the D2 line of alkali atoms is larger than that of the D1 line. Therefore, the absorption intensity of the D2 line of rubidium atoms is stronger than that of the D1 line.



Fig. 5. (Color online) Absorption spectra of alkali atom vapor cells at different temperatures. (a) The atom vapor cell with 4 mm diameter cavity. (b) The atom vapor cell with 5 mm diameter cavity. (c) The atom vapor cell with 6 mm diameter cavity.

3.2. Absorption spectra of alkali atom vapor cells with different silicon cavity diameters

The relationships between the intensities of the two absorption peaks at 780 and 795 nm of the atom vapor cells and the diameters of the silicon cavities under different heating temperatures are shown in Fig. 6. It can be seen that under the same heating temperature of the alkali atom vapor cells, the intensities of the absorption peaks at 780 and 795 nm of the rubidium atoms are almost unchanged with the change of silicon cavities from 4 to 6 mm, indicating that the intensities of the absorption peaks of the alkali atom vapor cells in this work are independent with the size of the silicon cavity.

3.3. Absorption spectra of alkali atom vapor cells with different RbN₃ filling amounts

The absorption spectra of alkali atom vapor cells with RbN₃ filling amounts of 125, 250, 375 and 500 μ g in the silicon cavities are compared, as shown in Fig. 7. It can be seen that the intensities of the two absorption peaks of the rubidium atoms do not change much with the increase of the filling amount of RbN₃. So the absorption peak intensities of the alkali atom vapor cells in this paper have almost no relationship with the filling amounts of RbN₃ at the same temperature.

It can be seen that when the heating temperatures of the alkali atom vapor cells are the same, the absorption peak intensities of rubidium atoms at 780 and 795 nm have no relationship with the sizes of silicon cavity and the filling amounts of RbN_3 from the absorption spectra of alkali atom vapor cells with different silicon cavity sizes and different filling amounts of RbN_3 . It can be obtained from formula (1) that

the absorption peak intensity of alkali atoms in the atom vapor cell is related to the atomic vapor density. Therefore, it can be considered that the alkali atomic vapor densities in the atom vapor cells are the same under the same heating temperature of the alkali atom vapor cells regardless of the sizes of the silicon cavity and the filling amounts of RbN₃. It can also be inferred that the rubidium atoms in the atom vapor cells reach a saturated vapor state. In order to verify the conjecture, the mass of Rb atoms and RbN₃ that need to be filled in the alkali atom vapor cells to reach the saturated vapor state is calculated.

The minimum rubidium content needed in the alkali atom vapor cell increases with the rising temperature when the rubidum atoms reach a saturated vapor state. It can be inferred from this that the rubidium atoms in the atom vapor cell reach a saturated vapor state at other temperatures below 140 °C when they reach a saturated vapor state at 140°C. The minimum rubidium content in each alkali atom vapor cell is calculated as the rubidium atoms reach a saturated vapor state at 140 °C (which is liquid now). Suppose that the mass of rubidium atoms in a single atom vapor cell is $m_{\rm Rb}$. The saturated vapor pressure $P_{\rm V}$ at temperature T (unit is K) is calculated according to the saturated vapor pressure model of rubidium atoms in liquid state^[18].

The formula for the saturated vapor pressure of rubidium atoms is^[19]:

$$\lg P_{\rm V} = 2.881 + 4.312 - \frac{4040}{T},\tag{3}$$

where P_V is the saturated vapor pressure in the atom vapor cell in Torr and *T* is the thermodynamic temperature in K.

The volume of the atom vapor cell can be expressed as



Fig. 6. (Color online) The relationship between the intensities of the two absorption peaks of the alkali atom vapor cells and the silicon cavity diameters. The heating temperatures of the alkali atom vapor cells are (a) 100 °C, (b) 120 °C, (c) 130 °C, (d) 140 °C.

$$V = \frac{1}{4}\pi\Phi^2 h, \tag{4}$$

where Φ is the diameter of the silicon cavity, and *h* is the thickness of the silicon wafer. Then according to the ideal gas state equation:

$$PV = nRT,$$
 (5)

calculate the number of moles of rubidium atoms at which the rubidium atomic vapor is saturated in the single alkali atom vapor cell (assuming rubidium atoms are all present as vapor).

Calculate the saturated vapor pressure of rubidium atoms at 140 °C according to Eq. (3).

$$P_V$$
 (140 °C) = 2.6 × 10⁻³ torr = 0.347 Pa. (6)

Substitute Eqs. (3), (4) and (6) into Eq. (5) to calculate n_0 (80 °C):

$$n_{0} (140 \ ^{\circ}\text{C}) = \frac{P(140 \ ^{\circ}\text{C}) V}{RT} = \frac{0.347 \times \frac{1}{4} \times 3.14 \times \Phi^{2} h}{8.314 \times 413.15} \text{ mol}$$

= 7.93 × 10⁻⁵ \Phi^{2} h mol. (7)

Calculate the mass of Rb or RbN_3 required in a single alkali atom vapor cell according to Eq. (7) (calculated with the relative atomic mass of ⁸⁷Rb):

$$m_{\rm Rb} (140 \ ^{\circ}{\rm C}) = n_0 M_{\rm Rb} = 7.93 \times 10^{-5} \Phi^2 h \times 86.909$$

= 6.892 × 10⁻³ $\Phi^2 h$ g, (8)

$$m_{\text{RbN}_3} (140 \ ^{\circ}\text{C}) = n_0 M_{\text{RbN}_3} = 7.93 \times 10^{-5} \phi^2 h \times 127.4879$$
$$= 1.011 \times 10^{-2} \phi^2 h \text{ g.}$$
(9)

The unit of the diameter and length of the alkali atom vapor cell is meter in the above formula.

For an alkali atom vapor cell with a silicon cavity diameter of 6 mm and a silicon wafer thickness of 1 mm, the mass of RbN_3 needed to be filled in the atom vapor cell is:

$$m_{\text{RbN}_3}$$
 (140 °C) = $n_0 M_{\text{RbN}_3}$ = 1.011 × 10⁻² $\Phi^2 h$ g = 3.6396 × 10⁻¹⁰ g.

It is calculated that the alkali atom vapor cell with the silicon cavity of 6 mm needs to be filled with a mass of at least 3.6×10^{-10} g of RbN₃ to make rubidium atoms reach a saturated vapor state at 140 °C. However, the mass of RbN₃ in the alkali atom vapor cell in this work is at least 125 µg, which is about 10⁵ times of the theoretical calculation value. Consequently, the rubidium atoms in the alkali atom vapor cell are sufficient. Therefore, when the alkali atom vapor cells are heated to a certain temperature, the rubidium atomic vapor pressures in the atom vapor cells are saturated. This indicates the rubidium atomic vapor densities are the same for all atom vapor cells with the same temperature. Consequently, the absorption peak intensities of the rubidium atoms at 780 and 795 nm are not variable with the change of the silicon cavity sizes and RbN₃ filling amounts.

4. Conclusion

In this work, chip-sized alkali atom vapor cells with different silicon cavity sizes and different alkali filling amounts

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Fig. 7. (Color online) Comparison of the absorption spectra of the alkali atom vapor cells with RbN₃ filling amounts of 125, 250, 375 and 500 μ g in the silicon cavities. The heating temperatures of the alkali atom vapor cells are (a) 110 °C, (b) 120 °C, (c) 130 °C, (d) 140 °C.

were prepared by MEMS technology. A batch of alkali atom vapor cells with high hermeticity were successfully fabricated by deep silicon etching and two anodic bonding processes, and the leakage rate could reach 1×10^{-9} Pa·m³/s. The guantitative filling of Rb atoms and buffer gas nitrogen in the atom vapor cell is realized by photolysis of RbN₃. A self-built absorption spectrum testing system is used to measure the absorption spectra of rubidium atoms in the alkali atom vapor cells with different silicon cavity sizes, different RbN₃ filling amounts and different temperatures. It is found that the absorption peak intensities of rubidium atoms in the alkali atom vapor cell become stronger with the increase of the temperature of the atom vapor cell, which is due to the increase of the rubidium atomic vapor density in the atom vapor cell with the rising temperature. The absorption peak intensities of rubidium atoms in the alkali atom vapor cells have almost little dependence on the size of the silicon cavity and the filling amount of RbN₃ under the same heating temperature. After theoretical calculation of the minimum mass of RbN₃ required for rubidium atoms reaching the saturated vapor state at 80 °C in the alkali atom vapor cell, it is found that the amount of rubidium atoms in the alkali atom vapor cell is large enough for the saturated vapor state to be reached at each temperature. Consequently, the rubidium atomic vapor densities are the same for all atom vapor cells with the same temperature. Therefore, the absorption peak intensities of the rubidium atoms at 780 and 795 nm are not variable with the change of the silicon cavity size or RbN₃ filling amount. This paper provides a reference for the design and fabrication of high quality chip-sized alkali atom vapor cells.

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