Sensors and Actuators A 167 (2011) 594–601

Contents lists available at ScienceDirect

Sensors and Actuators A: Physical

journal homepage: www.elsevier.com/locate/sna

Microfabrication of cesium vapor cells with buffer gas for MEMS atomic clocks

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ARTICLE INFO

Article history:
Received 26 July 2010
Received in revised form
22 December 2010
Accepted 22 February 2011
Available online 2 March 2011

Keywords:
Anodic bonding
Alkali-vapor cells
Buffer gas
MEMS atomic clock
Optical absorption spectroscopy

ABSTRACT

This paper reports on the Si-glass anodic bonding process to fill micro Cs vapor cells with a buffer gas (Ar or Ne) at a controlled pressure (up to 20 kPa), which is one of the technological key steps to fabricate Cs vapor cells for miniature atomic clocks. In the atmosphere of these gases, the applicable bonding voltage was not high enough to achieve strong bonding because of the electrical breakdown caused by ionization of the gas. To improve the bonding quality, an original two-step anodic bonding method was proposed. The first step of the anodic bonding, which intends to pre-seal the gas in microcells, is carried out in the presence of a buffer gas by applying a voltage lower than the breakdown voltage. Subsequently, the second bonding is performed in air at sufficiently high voltages to improve the sealing quality.

By employing optical spectroscopy, it was demonstrated that the cells maintain the buffer gas at an appropriate pressure for atomic clock operation. The accelerated aging tests show that Cs vapor as well as the buffer gas remained in the cells without any significant change in the pressure, which allow us to estimate the lifetime of the cells to be at least 3 years. Further CPT experiments revealed that the buffer-gas pressure change is less than 6.13×10^{-4} kPa throughout the aging test at 125 °C for more than 3 weeks. These results show that these microcells are appropriate for applications to atomic frequency references.

1. Introduction

Atomic clocks provide enhanced frequency accuracy and long-term frequency stability, compared to quartz-crystal oscillator-based clocks. Recently, highly miniaturized, battery-powered chip-scale atomic clocks (CSAC) have attracted attention because of their great potential in civil and military applications ranging from telecommunication systems to global positioning as well as synchronization of communication networks. The first architecture of CSAC, employing the principle of coherent population trapping (CPT) [1,2], has been demonstrated by Kitching's group at NIST [3]. Furthermore, there have been considerable efforts to achieve CSACs carried out by several other research groups in USA [4] as well as industrial companies [5–7]. The great advantage of CPT clocks compared to classical Rb vapor cell clocks is that they use an all-optical interaction to probe the atomic resonance avoiding the use of a microwave cavity. This allows a significant reduction of the clock volume and the power consumption.

The heart of a CSAC consists of a micro alkali-vapor cell illuminated by a high-frequency modulated laser beam. The light passing through the cell is collected by a photodiode which delivers a signal used to lock the frequency of a local oscillator on the ground state hyperfine resonance of the atoms.

Most of existing microcell structures are fabricated by conventional microfabrication technologies including photolithography, dry or wet etching to fabricate cavities in a Si wafer, and subsequent anodic bonding of thin glass wafers to both surfaces of the Si wafer [3,8–10]. Many methods have been used to fill the cells with Cs (or Rb) so far. Knappe et al. proposed the method which utilizes a chemical reaction of an alkali chloride and the reducing agent included in a cell to obtain required Cs liquid before the cell is sealed [3]. Later, they reported the modified process where Cs gained by the former method is directly injected in the cell in order to improve the long-term clock frequency stability [10]. However, in both cases, the presence of volatile alkali metal complicates the operation of anodic bonding, performed at high temperature. To overcome this technological problem, several solutions to incorporate Cs after the cell-sealing have been proposed and investigated. Liew et al. [11] reported a Cs filling method where cesium azide thin films, which are chemically stable up to 450 °C, are deposited in the cavities of microcells and then photodecomposed into Cs and N_2 using ultraviolet irradiation after sealing of the cells by anodic bonding in vacuum. Gong et al. [12] proposed another method in which Cs is obtained by electrolysis of Cs-enriched glass placed on the inner surface of a sealed microcell. On the other hand, we recently proposed a simple Cs filling method where Cs vapor is generated by locally heating a Cs dispenser with a high-power laser source after

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doi:10.1016/j.sna.2011.02.039

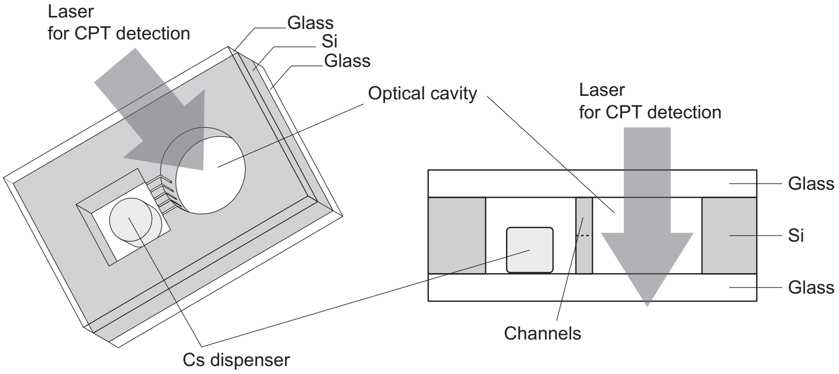

Fig. 1. Design of a microcell; (a) perspective view and (b) cross section.

the sealing of cells [13]. The dispenser, consisting of the cesium chromate and Zr–Al alloy mixture, exhibits a high chemical stability up to 500 °C while it is activated at higher temperatures (~700 °C) to generate Cs vapor. We demonstrated that the Cs dispensers do not affect the quality of anodic bonding [9] and that pure Cs is generated by laser activation [8,9]. However, several technological steps need to be studied for the application of this technology to atomic clocks. In particular, microcells for CSACs are generally filled with the buffer gas (in most cases, N_2, Ne or Ar) at a required pressure in order to avoid the CPT signal broadening induced by wall-collision of alkali atoms [14,15]. Moreover the pressure of the gas is required to be stable because the changes in the buffer gas pressure induce clock frequency variations with time, which affect the long-term frequency stability. In our microcells, Ne or Ar is employed because they are not affected by the Cs dispenser, unlike N_2 which was found to be absorbed by the dispenser [16]. According to the literature [17], the sensitivity of the clock frequency with respect to changes in the buffer gas pressure is -1.61×10^{-7}/kPa and 5.41×10^{-7}/kPa for Ar and Ne, respectively. Therefore, to achieve a long-term relative frequency stability of 1×10^{-11} at 1 day, the variation of the gas pressure for a day should be within 1.84×10^{-5} kPa for Ne and 6.21×10^{-5} kPa for Ar, respectively.

In Section 2, we describe the steps of the microcell fabrication and the technological issues for fabrication. We focus our investigation on the anodic bonding process to fill microcells with the buffer gas at a controlled pressure as well as to achieve high sealing quality in the presence of gas. When the anodic bonding is performed in the atmosphere of Ar or Ne, the process may be disturbed by the electrical breakdown caused by gas ionization, as like the case of the anodic bonding in N_2 atmosphere [18]. Therefore, to obtain an insight into the electrical breakdown phenomena for Ar and Ne, the breakdown voltage was measured in the wafer bonding chamber filled with Ar or Ne at required pressures (up to 20 kPa). According to the results of the breakdown voltage measurements, a two-step anodic bonding method was proposed to seal microcells with the buffer gas.

Finally, in Section 3, we report the results of the optical and CPT-resonance spectroscopy of microcells to examine the quality of the cells. Accelerated aging tests were performed at temperatures higher than the clock operation temperature for a few weeks to a few months in order to study the variation and aging of the inner atmosphere of the microcells.

2. Microfabrication of atomic vapour cells and technological issues

2.1. Process flow of fabrication

The cells are formed by sandwiching a Si wafer with two though-hole cavities between two borosilicate glass wafers by anodic bonding (Fig. 1). The diameter of optical cavity is 2 mm and the size of dispenser cavity is 1.65 mm × 1.65 mm. The optical cavity is used for CPT detection, whereas the dispenser cavity is used to store a Cs-dispenser pill supplied by SAES Getters. These cavities are connected through filtration channels to avoid the contamination from the Cs dispenser during its activation process. The inner thickness of the cell (the optical path length for CPT detection) is one of the important parameters determining the short-term relative clock frequency stability. In our case, the ultimate objective short-term frequency stability is 6×10^{-10} at 1 s. According to the literature reported by Kitching et al. [19], this can be achieved with the microcell with a thickness of around 1 mm or more. Therefore, we employed a Si wafer with a thickness of 1.4 mm, which will be sufficient to reach our objective.

For the fabrication of microcells, we essentially follow the process developed for the cells without the buffer gas, reported in Ref. [9]. A (1 0 0)-oriented p-type Si wafer of 4″ (1.4-mm thick) and two borosilicate glass wafers (0.5-mm thick) are used to fabricate microcells. The both surfaces of the wafers are polished prior to the process. First, an array of the optical and Cs dispenser cavities (through holes) and the surface filtration channels (trenches) are patterned on Si wafer by photolithography and etched in a Si wafer by the deep reactive ion etching (DRIE, Fig. 2a). Fig. 3a shows the cross-sectional SEM images of the Si cavity after DRIE. The surface of etched Si, especially the portions near the top and bottom ends of the cavities contained small defect-holes (seen as the white patterns). As is seen in the magnified image of the defect-portion (Fig. 3b and its inset), the surface was covered with the non-uniform and scallop-shaped defects. These defects could trap Cs atoms as well as the contaminants which may react with Cs. Moreover, it should be noted here that fluorocarbon passivation layers resulting from DRIE process may also affect the chemical stability of Cs vapor because the fluorocarbons are known to react with alikali metals to form alkali fluorides [20,21]. Therefore, to remove the defects on the sidewall surface along with the fluorocarbon deposits, the Si wafer is immersed in KOH aqueous solution (41 wt.%, 55 °C) for 30 min. Fig. 3c and d shows the SEM images of the sample after the KOH treatment. As is seen in these images, the surface defects were almost removed without any significant changes in the dimensions of cell-cavities. The magnified SEM images (Fig. 3d and its inset) show that the scallop-shaped micro-defects on the surface were totally polished and the crystal plans of Si start to appear on the surface. From this result, we consider that the fluorocarbon layers which were on the surface of the etched sidewalls were also totally removed by this process.

Followed by the etching of cell-cavities in a Si wafer, the wafer is anodically bonded to a glass wafer to obtain the Si/glass structure (Fig. 2b). Prior to the anodic bonding, the cavity-etched Si wafer and the glass wafer are cleaned in piranha solution (mixture of H_2O_2 and H_2SO_4, 1:4), and rinsed in deionized water. The wafers are then