Low-Temperature Alkali Metal Production Utilizing Scalloped Silicon Grooves for Microfabricated Alkali Vapor Cells

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INTRODUCTION
Microfabricated alkali vapor cells (hereafter MEMS cells) are in use for atomic-based instruments like atomic clocks or atomic magnetometers [1]. MEMS cells are a key component of atomic clocks that is based on Coherent Population Trapping (CPT) and generally fabricated by etching of a Si and anodic bonding. To fill MEMS cells with alkali metal, a wafer-level technique based on alkali-metal azides (CsN$_3$ or RbN$_3$) is a promising approach; however, prolonged UV-exposure was necessary to observe alkali metal [2]. Alternatively, an activation of the commercially available Cs dispenser [3] or alkali metal source tablet [4] have been proposed, which has potential for speeding up the Cs production. However, these Cs dispenser sealed in the cell might affect the frequency stability and degrade the aging performances.

In this paper, we report the different approach using Si groove with multiple re-entrant structures (i.e. scalloped patterns) to produce Cs. This approach enables effective thermal decomposition of CsN$_3$ on Si substrate at low temperature. The potential in atomic clock applications is also discussed.

METHOD OF CS PRODUCTION
Thermal decomposition of CsN$_3$ on Si or glass substrate requires high-temperature heating (around 550 °C). For solving this problem, the authors demonstrated that the CsN$_3$ crystal assembled on porous alumina structure enable to produce atomic Cs at around 300 °C [4]. The newly proposed Cs production method employs Si grooves with micro-size scalloped pattern that imitates porous alumina structure by Si structure as shown in Fig. 1. Si groove with scalloped patterns is fabricated by a sequence of isotropic and anisotropic deep reactive ion etching (DRIE) as shown in Fig. 2. Unlike the typical small scalloped pattern for high aspect ratio Si microstructures, the process parameters of isotropic etching are mainly adjusted to obtain large scalloped pattern. When an aqueous solution of CsN$_3$ pipetted in the grooves and dried, the recrystallized CsN$_3$ are assembled on the micro-size scalloped patterns. After an encapsulation in the MEMS cell, the sample is heated by a hotplate, and Cs and N$_2$ are injected to the CPT cavity (Fig. 3).

MEMS CELL FABRICATION
The wafer-level fabrication of MEMS cells shown in Fig. 4 starts with, (a) Cr deposition on a Si substrate and patterning to form the Cr mask, (b) DRIE etching of the top side to fabricate microchannels and two cavities, (c) bonding to bottom glass and inserting the sample, (d) sealing the cells by sequential plasma activated bonding [5], and finally (e) heating the sample to produce Cs.

RESULTS AND DISCUSSIONS
To confirm a potential of scalloped Si groove for Cs production, thermal decomposition of CsN$_3$ deposited on the Si substrate was investigated. During the heating in a vacuum chamber, time course of N$_2$ gas pressure as an indicator of decomposition was monitored. As shown in Fig. 5, the N$_2$ gas pressure obtained by the sample of scalloped Si grooves showed a similar trend with that of porous alumina structure [4], while the sample of Si grooves without scalloping showed the low efficiency. Thus the thermal decomposition of CsN$_3$ in scalloped Si grooves enables effective Cs production with similar uses as porous alumina.

Figure 6 shows a top view of the fabricated cells utilizing the scalloped Si grooves. In the present case, a metallic color of Cs was visible at 315 °C for about 15 min. Furthermore, spectroscopy measurement was carried out to demonstrate an ability to MEMS atomic clocks. The short-term frequency stability is measured to be $5 \times 10^{-11}$ at an integration time of 1 sec. This stability compares well to the performance of previously reported ones [2-4]. Improvement of the stability could be obtained by further optimizing process parameters on thermal decomposition.

CONCLUSIONS
We have presented the low-temperature fabrication of MEMS cells utilizing scalloped Si grooves for effective Cs production. The Cs-filled MEMS cells made in the present fabrication is applicable in MEMS atomic clocks. To confirm the long-term suitability, the evolution of the frequency stability along time should be investigated in the future.

REFERENCES
Figure 1. SEM image of (a) Si substrate with grooves with scalloping (left side: top view, right side: close-up view), and (b) scalloped Si grooves before/after deposition of CsN$_3$ crystal (cross-section view).

Figure 2. Fabrication of the micro-size scalloped Si groove by DRIE process. (a) Cr for the hard mask was deposited, and (b) non-grooved region was defined by lithography for subsequent Cr etching (c). In order to form multiple scallops in the thickness direction, steps from (d) to (h) are repeated: (d) anisotropic etching and sidewall protection, (e) passivation removal at the bottom of groove, (f) isotropic etching, (g) sidewall protection, and finally (h) passivation removal at the bottom of re-entrant structure.

Figure 3. Schematic illustration of the MEMS cell presented in this work. Thermal decomposition of CsN$_3$ produces Cs and N$_2$ buffer gas in-situ. The size of the sample cavity is 8 mm × 8 mm. Two cavities are connected via microchannels to avoid the contamination during thermal decomposition process.

Figure 4. Wafer-level process of the MEMS cells. In the step (4), two cavities (through holes) and microchannels were fabricated by the optimized single-step of DRIE. At the step (6), Si substrate with CsN$_3$ was encapsulated by sequential plasma activated bonding in high vacuum at a temperature of 250 °C and applying a potential of 250 V.

Figure 5. Measured N$_2$ gas pressure for different types of container. Two samples to compare with the scalloped Si grooves were also prepared as references. One was the Si grooves without scalloping, and the other was the porous alumina with 60 μm pores.

Figure 6. Close-up view of the wafer-level fabricated MEMS cells before/after the heating process. The existence of Cs produced in the cell was also confirmed by optical absorption spectroscopy.