Perforated hollow-core optical waveguides for on-chip atomic spectroscopy and gas sensing

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A hollow-core waveguide structure for on-chip atomic spectroscopy is presented. The devices are based on Anti-Resonant Reflecting Optical Waveguides and may be used for a wide variety of applications which rely on the interaction of light with gases and vapors. The designs presented here feature short delivery paths of the atomic vapor into the hollow waveguide. They also have excellent environmental stability by incorporating buried solid-core waveguides to deliver light to the hollow cores. Completed chips were packaged with an Rb source and the $F = 3 \rightarrow F' = 2, 3, 4$ transitions of the D2 line in ^{85}Rb were monitored for optical absorption. Maximum absorption peak depths of 9% were measured. © 2016 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4945092>]

Micro-scale atomic spectroscopy devices which rely on the interaction of light with atoms and molecules in the gas phase are useful in many areas. Such devices may also be used for gas sensing applications including toxic gas monitoring¹ and pollution control.²

Recently, much effort has gone into miniaturizing atomic spectroscopy platforms³ in which optical beams probe the energy states of atoms. Atomic spectroscopy with alkali vapor is particularly promising because it has been used to demonstrate tunable photon pulses⁴ and can be used for observation of Rydberg states⁵ and quantum interference effects in linear and nonlinear optics.^{6,7} Successful micro-scale atomic spectroscopy systems include atomic cladding waveguides,⁸ anodically bonded cells,⁹ silica hollow-core fibers,^{10,11} polymer-based hollow-core fibers,¹² and hollow-core waveguides on silicon.¹³ Several of these atomic spectroscopy platforms require that vapor be transferred from a reservoir to an active region, where it interacts with an optical probe beam. In these designs, as well as in other sensing and spectroscopy applications, minimizing the length between the reservoir and interaction region is beneficial because diffusion-limited transport of atoms through micron-scale channels can be quite slow. Having the gas reach the active region quickly also allows for faster measurements and reduces the possibility of contamination or undesirable interactions and loss of atoms in the transfer space.

In order to optimize the performance along these lines, we have implemented a device design for a self-contained hollow waveguide platform. Instead of opening the hollow cores from either extremity, several holes are opened immediately over the top of the waveguide. Similar methods have been used in other waveguides and optical fibers.¹⁴ Gas or vapor can be supplied from a single reservoir encompassing the entire length of the hollow-core waveguide instead of two reservoirs located at both ends of the waveguide. Optical

signals are directed to and from the hollow waveguide using solid-core waveguides. Moreover, since any contamination on the surface of a ridge waveguide is catastrophic to transmission, the design incorporates buried channel waveguides (BCWs). There are several advantages to this platform. First, the distributed access along the hollow waveguide leads to almost immediate loading of gas through the top holes. This reduces transport or diffusion problems and minimizes the risk of contamination. In cases where wall-vapor interactions are detrimental to device operation, such as with alkali vapor,¹⁵ a minimal path length to the active region is ideal. Second, the single reservoir simplifies the loading and packaging. Finally, using BCWs makes the platform less susceptible to environmental contaminants or water absorption.

One concern to this design is additional transmission loss due to the perforations in the hollow waveguide. Simulations were run using FIMMWAVE to explore the impact of these top holes on transmission. These simulations show that the loss increases with increasing hole diameter. Using typical waveguide geometries ($5 \times 12 \mu\text{m}$ cross-sections), these simulations predict an optical loss of 0.3 dB per hole using $5 \mu\text{m}$ square holes. This provides the necessary guidance in determining the hole density for a particular hollow core length that balances overall transmission and optimized vapor loading.

Microfabrication of the proposed design was done on 100 mm diameter silicon wafers, which were first sputtered with alternating SiO_2 and Ta_2O_5 layers to create anti-resonant reflecting optical waveguide (ARROW) layers.^{16,17} These layers have refractive indexes of 1.46 and 2.275 and precise thicknesses of $184 \pm 2 \text{ nm}$ and $110 \pm 1 \text{ nm}$, respectively, so that the final 3-period stack is highly reflective, facilitating guiding in the low-index solid and hollow waveguide cores. Next, a sacrificial core and the solid core of the BCWs were deposited and patterned with SU8–10 as shown in Figure 1(a). AZP4620 photoresist was spun onto the wafer and developed to expose the top half of the cores and the BCW area. The wafer was then placed in an electron beam evaporator and 80 nm of nickel were deposited. After patterning and

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FIG. 1. Fabrication process for perforated hollow-core waveguide atomic spectroscopy chip. The core and BCW are patterned with SU-8 (a); a pedestal is etched (b); the top oxide is deposited, patterned, and etched to create holes over the core ((c) and (d)). Finally, a reservoir is attached.

lift-off, the nickel served as a mask for the subsequent anisotropic plasma etching steps. The bottom layers were etched using a reactive ion etcher (RIE) with an inductively coupled plasma (ICP) RF generator.

Once the SiO_2 and Ta_2O_5 layers were removed, another $4\text{ }\mu\text{m}$ of the silicon substrate was etched using a deep RIE etch. Finally, anisotropic silicon etching was used to remove any surface roughness created by the amplification of microscopic defects during the other etch processes. We minimize the surface roughness in order to maximize the quality of the top oxide layer. These etching steps effectively put the core on a pedestal which greatly increased its final transmission.¹⁸ At this point, the wafer looked like Figure 1(b). A $6\text{ }\mu\text{m}$ thick top oxide layer was deposited by PECVD at $250\text{ }^\circ\text{C}$. In order to expose the sacrificial SU-8 core, the wafer was coated with a 100 nm layer of chrome in the E-beam evaporator. The holes were patterned in AZ4620 and the wafer was

placed in chrome etchant to open up the holes. The holes were wet etched in hydrofluoric acid until they reached a depth of $4\text{ }\mu\text{m}$. The chrome protected the top of the core, while the AZP4620 protected the sharp edges where the chrome was weaker. After the wet etch, the wafer was once again etched in the Trion ICP-RIE using the same recipe used for the bottom layer etching. Wet etching did a very little damage to the photoresist mask but is isotropic and therefore widened our holes. On the other hand, ICP-RIE etching was very anisotropic, thus maintaining the aspect ratio but was very damaging to the mask. The dual-step etching process assured a balance between the hole dimension and mask durability. The growth and patterning of the top oxide are shown in Figures 1(c) and 1(d).

At this point, the entire SU-8 core was removed in a piranha etch ($1:1\text{ } 50\% \text{ H}_2\text{O}_2: 98\% \text{ H}_2\text{SO}_4$) at $100\text{ }^\circ\text{C}$. The wafer was left in this acid solution for 3 h, after which it was rinsed thoroughly in deionized water to complete the fabrication. The device now consists of a hollow core waveguide coupled to an SU-8 BCW at each extremity. An SEM image of the chip surface is shown in Figure 2. Reservoirs were attached as shown in Figure 1(e), readying the device for loading with the vapor or gas of interest.

Atomic spectroscopy with rubidium vapor was used to evaluate the device performance. Note that rubidium represents an extreme case for this platform in terms of material selection and required packaging. Only a short list of materials are compatible with Rb because of its reactivity. Oxidized copper tubing was attached to the chips with epoxy (Aremco-Bond 2310). The chips were loaded with rubidium in an inert gas glovebox by placing a $\sim 1\text{ }\mu\text{l}$ piece of solid Rb into the tubing, evacuating it to $<1\text{ mTorr}$, and then hermetically sealing the tubing using a cold weld crimping tool (CHA Industries POD-375).

The loaded devices were held on a platform heated to $85\text{ }^\circ\text{C}$ and monitored with a 780 nm laser [ThorLabs ITC4001] tuned to sweep across the Rb D2 absorption line transitions. The loss for the structure is 10.9 dB/cm for light polarized in-plane and 421.3 dB/cm for light polarized out of the chip plane. The testing components used are illustrated in Figure 3. The collimated light was focused and coupled into an optical

FIG. 2. SEM image of a finished chip. The BCW sits on a wider pedestal (top of the image) and is aligned to the perforated hollow-core waveguide below.

FIG. 3. Diagram of atomic spectroscopy setup. The copper stub is shown transparent for clarity.

FIG. 4. Absorption spectra from loaded devices tested at 75 °C, 85 °C, and 95 °C.

fiber before guiding through the on-chip waveguides and finally hitting a photodetector [Thorlabs PDA36A]. The chips were characterized by the optical depth of the deepest Doppler broadened absorption peak extracted from the $D2^{85}\text{Rb } F=3 \geq F'=2, 3, 4$ transitions. The collected spectra are compared to a commercial bulk cell reference spectrum. Absorption was visible within hours of heating the chip since rubidium vapor directly entered the active hollow core through the top perforations. Representative spectra are shown in Figure 4 when a chip was heated to three different temperatures.

The four dips in transmission shown in Fig. 4 match the theoretical expectations for the sum of the twelve hyperfine peak profiles of the $D2$ line in a natural mixture of ^{85}Rb and ^{87}Rb . The depths of these absorption peaks can be predicted using an equation for the absorption coefficient which incorporates the Voigt profile, defined as

$$\alpha_{\text{theory}} = k \times \frac{C_{\text{trans}} N}{D} \times \frac{V^I(x)}{\hbar \epsilon_0}, \quad (1)$$

where k is the wavenumber of the probe beam, C_{trans} is the transition strength, N is the atomic density, D is the degeneracy of the ground state of the isotope in question, and $V^I(x)$ is the imaginary part of the Voigt profile.¹⁹ This absorption coefficient can then be used to find the theoretical transmission using the following equation:

$$t_{\text{theory}} = \exp(-\alpha_{\text{theory}} \times L), \quad (2)$$

where L is the interaction length, which in this case is 4 mm. To relate this expected transmission profile to our measured spectra, atomic density must be calculated for a given temperature. The atomic density term in Equation (1) is further broken down to

$$N = \frac{133.323 \times p}{k_b T}, \quad (3)$$

where p is the Rb vapor pressure in Torr (for liquid phase Rb) with the following equation:²⁰

$$\log_{10} p = -94.05 - \frac{1961}{T} - 0.038T + 42.58 \log_{10} T. \quad (4)$$

The measured absorption in Fig. 4 is about 6% of what the model predicts. This discrepancy can be attributed to several

factors. First, the investigations of the guiding mode profile at the chip output indicate that as much as 50% of the output light through the device may be transmitted through the cladding layers instead of the hollow core. This light would not have a chance to interact with the atomic vapor. Additionally, various contamination and transport delays due to wall-vapor interaction can slow the rate at which the Rb enters the core or reduces its viable lifetime.²¹ Changes to the loading process or the addition of anti-relaxation wall coatings or buffer gases can greatly mitigate the transport problems and increase the overall absorption,²² but even the coated cells require a “ripening” time before proper operation.²³ Since Rb is taken as an extreme case, such changes may not be necessary in applications monitoring less reactive gases.

In summary, we have demonstrated that a perforated single-reservoir ARROW platform can be used for atomic vapor spectroscopy over extended periods of time. The platform shows great promise for further miniaturization and integration of atomic vapor cells and other sensing devices into silicon-based processes. When testing the platform with Rb, absorption is evident within a few hours and lasts several days at elevated temperatures. The performance can be further enhanced by improving the transmission properties of the SU-8 BCW and hollow core. Improvements can also be made by further optimizing the bottom layers or by decreasing the loss experienced at the hole sites above the core.

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