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Enhancement of ARROW Photonic Device Performance via Thermal Annealing of PECVD-based SiO₂ Waveguides

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Abstract

Silicon-based optofluidic devices are very attractive for applications in biophotonics and chemical sensing. Understanding and controlling the properties of their dielectric waveguides is critical for the performance of these chips. We report that thermal annealing of PECVD-grown silicon dioxide (SiO₂) ridge waveguides results in considerable improvements to optical transmission and particle detection. There are two fundamental changes that yield higher optical transmission: (1) propagation loss in solid-core waveguides is reduced by over 70%, and (2) coupling efficiencies between solid- and liquid-core waveguides are optimized. The combined effects result in improved optical chip transmission by a factor of 100–1000 times. These improvements are shown to arise from the elimination of a high-index layer at the surface of the SiO₂ caused by water absorption into the porous oxide. The effects of this layer on optical transmission and mode confinement are shown to be reversible by alternating subjection of waveguides to water and subsequent low temperature annealing. Finally, we show that annealing improves detection of fluorescent analytes in optofluidic chips with a signal-to-noise ratio improvement of 166x and a particle detection efficiency improvement of 94%.

Index Terms

Annealing; anti-resonant reflecting optical waveguides (ARROWs); biophotonics; integrated waveguides; liquid core waveguides; optofluidics; PECVD; silicon dioxide

I INTRODUCTION

Silicon-based micro- and nanophotonic devices are extremely attractive for chip-scale integration of infrared applications such as telecommunications, IR sensing and quantum computing [1]–[3]. Because silicon absorbs in the visible spectrum, it cannot be used as a waveguide material for many established techniques in biological and chemical sensing. Fortunately, many dielectric materials such as silicon's native oxide as well as other oxides and nitrides have good optical properties in the visible range and can be integrated on a silicon platform using established fabrication methods [4].

Recently, SiO₂ has been extensively implemented in optofluidic sensing chips to form optical waveguides designed for operation at visible wavelengths that are compatible with fluorescence biosensing applications. As a material, SiO₂ is a key component of optofluidic sensing chips for forming anti-resonant reflecting optical waveguide (ARROW) layers [5], [6], liquid-core waveguide walls [7], as well as excitation and collection solid-core ridge waveguides [8]. Many recent implementations of ARROW chips rely heavily on an orthogonal excitation/collection geometry for fluorescence detection [9]. As such, optimization of these SiO₂ ridge waveguides is of paramount importance and has been extensively studied [10]–[12]. Here, we show that a distinct upper layer (DUL) with increased refractive index in the SiO₂ ridge waveguides is formed during fabrication. This layer causes significant degradation of optical chip quality, concerning both power transmission and mode shape.

We demonstrate that low temperature thermal annealing eliminates the DUL and its effects. After annealing, silicon dioxide ridge waveguides have dramatically increased confinement within the core, leading to substantially lower optical propagation losses and improved solid-to liquid-core waveguide coupling efficiencies. Finite difference method (FDM) simulations support experimental results showing that optical field localization within the DUL causes high loss, both in propagation and waveguide to waveguide coupling. Repeated cycles of boiling (in water) and thermal annealing of silicon dioxide waveguides suggest that water absorption into the porous oxide is the cause of the DUL. Finally, we report dramatic signal improvements for fluorescence particle detection in orthogonal beam geometry produced by thermal annealing of optofluidic detection chips.

II CHIP DESIGN, FABRICATION, AND PROCESSING

Optical waveguides were designed and fabricated using the Anti-Resonant Reflecting Optical Waveguide (ARROW) concept [5]. ARROW structures are based on one or more layers fulfilling the anti-resonance condition to form a reflective mirror. When these layers are placed beneath a waveguide core on a high index substrate, optical radiation modes can be well confined in a low refractive index core material [13], [14]. Liquid-filled ARROWs have recently been used for reconfigurable optical devices [15]–[17].

ARROW optofluidic chip fabrication integrates many standard CMOS procedures [18]. Briefly, a total of six SiO₂ (refractive index: $n = 1.47$) and Ta₂O₅ ($n = 2.107$) anti-resonant layers were alternately deposited on a silicon wafer with 265 and 102 nm respective

thicknesses. This ARROW stack was designed for broadband low-loss transmission of visible and near-IR light, centered at 660 nm [13]. Standard SU-8 lithography was used to define a 12 μm wide and 5 μm tall sacrificial core. A silicon pedestal for waveguide/substrate isolation was then created by deep reactive ion etching (DRIE) [12]. Plasma enhanced chemical vapor deposition (PECVD) of SiO_2 was used to create a 6 μm thick overcoating [10]. Another SU-8 lithography step—in conjunction with DRIE—was used to define 4 μm wide ridge waveguides by selectively etching away 3 μm of the top SiO_2 layer. The sacrificial core was exposed and removed using piranha ($\text{H}_2\text{SO}_4\text{:H}_2\text{O}_2$) etching [19]. It is important to note that this core removal step takes approximately 120 hours in a hot (90°C) bath of etch solution. During this procedure, liquid can diffuse through the semi-porous PECVD SiO_2 layer, leading to a DUL which will exhibit a higher refractive index than an SiO_2 layer with only air in the pores. Fig. 1 shows the cross section of a solid-core ridge waveguide fabricated with this approach. The SEM image in Fig. 1 suggests a non-uniform top SiO_2 layer, featuring a distinct upper layer as illustrated schematically. After fabrication, the waveguides were subjected to repeated cycles of thermal annealing and immersion in liquid to elucidate the nature and origin of this layer.

Low temperature thermal annealing was performed in an annealing furnace at atmospheric pressure by placing diced chips from a wafer onto a ceramic plate (Thermo-Fisher). The furnace was programmed to ramp from room temperature to 300°C over 4 hours (to avoid rapid thermal expansion and cracking of SiO_2 layers), maintain a 300°C temperature for 8 hours, and ramp down to room temperature over no less than 4 hours (subject to dissipative cooling).

All optical characterization experiments were performed through butt-coupling of laser light from a fiber with 4.3 μm mode field diameter and 0.12 NA at 633 nm into the solid-core ARROW. Transmission and optical mode imaging experiments were performed by collecting light from output waveguide facets through an objective, either onto a photodetector or mode imaging camera (Photon, Inc.), respectively.

III OPTICAL CHARACTERIZATION

A. Optical Confinement and Loss in Solid-core Waveguides

First, we determined the effects on thermal annealing has significant effects on the properties of PECVD-grown solid-core SiO_2 ridge waveguides. As seen in Fig. 2a, as-grown ARROWs exhibited considerable lateral divergence of butt-coupled laser light, spreading quickly over a distance of $\sim 250\mu\text{m}$ before being laterally confined by the silicon pedestal. When annealed following the procedure described above, lateral optical confinement underneath the 4 μm wide ridge is dramatically improved. In order to quantify the increase in lateral confinement upon annealing, the optical mode profile was imaged at the waveguide facet. As seen in Fig. 2b, an as-grown ARROW chip has poor lateral confinement—34% within the ridge core, after 1 cm of propagation. Note that the mode intensity was integrated in the vertical (y) direction to obtain the plots seen in Fig. 2b. Upon annealing, the confinement of the ARROW increases to 84% within the ridge core, a nearly 2.5-fold improvement. There is an apparent discrepancy in core confinement between Fig. 2a and Fig. 2b as-grown samples. This is explained by the fact that the loss of widely spread modes is higher than that of the

fundamental ridge modes and thus appears more prominently in the top-down view of Fig. 2a. In addition, the images in Fig. 2a cover a propagation distance of $\sim 250 \mu\text{m}$, while the mode images in Fig. 2b were taken at 1 cm length, where a significant portion of the unconfined light had already been lost.

In order to obtain the waveguide loss for the light confined within the $4 \mu\text{m}$ ridge, two adjacent solid-core waveguides were separated and subjected to a cut-back analysis. Note that output optical intensity was focused through an iris, collecting intensity propagating within the core and $10 \mu\text{m}$ to either side. The results displayed in Fig. 2c show that the annealed ARROW had an optical loss coefficient ($\alpha_A = 0.47 \text{ cm}^{-1}$) over three times lower than that of an as-grown ARROW ($\alpha_{AG} = 1.63 \text{ cm}^{-1}$). We also note that as we are selecting the intensity within the core for this measurement, the as-grown sample exhibits the lower limit of its loss coefficient. If included, the more divergent/lossy light outside of the ridge core would contribute to a higher loss value.

Finite difference method (FDM) optical modeling was used to further investigate the difference between as-grown and annealed solid-core waveguides. The sample dimensions were extracted from the SEM cross-section of a solid-core waveguide shown in Fig. 3a. While the silicon substrate, ARROW layers, and ridge waveguide profile matched their design specifications, the presence of a DUL (designated 4) is readily apparent and follows the ridge profile with a thickness of $\sim 2 \mu\text{m}$. Energy-dispersive X-ray (EDX) analysis confirmed the presence of this layer, and showed that the DUL had markedly higher oxygen content. Specifically, the bulk oxide had an expected 2:1 ratio of oxygen to silicon while the DUL had a 2.8:1 ratio of oxygen to silicon. This increase in oxygen content can be explained by water absorption. In accordance with literature data concerning water absorption into a porous SiO_2 layer, the DUL was set to have a higher refractive index ($n_4 = 1.47$) than the lower SiO_2 layer ($n_3 = 1.46$) [20]. With this inclusion, modal simulations of as-grown waveguides match experimentally determined patterns, as seen in Fig. 3b (left) for a 3 mm long ARROW waveguide. Furthermore, when the mode profile is simulated with a homogeneous SiO_2 layer ($n_3 = n_4 = 1.46$), the fundamental ridge mode is primarily excited. Fig. 3b (right) shows that after annealing, the ARROW chip has a mode matching that of a ridge waveguide fundamental mode. The simulated $1/e^2$ fundamental mode width for the given waveguide structure— $3.6 \mu\text{m}$ —is in very good agreement with the measured width— $3.3 \mu\text{m}$. Furthermore, the simulated modes presented in Fig. 3 have confinement factors (relative intensity within the ridge width) of 43% and 89% for as-grown and annealed, respectively. This is in excellent agreement with the experimentally observed confinement factors of 41% and 89%. We also note that even though the sample presented in Fig. 3 is different than that of Fig. 2, there is relative agreement between the data.

There is some variability amongst as-grown ARROW chip performances due to fabrication inhomogeneities as well as the uncontrolled nature of water absorption, which inherently gives rise to varying DUL dimensions. Although the as-grown structure in Fig. 3b exhibits no fundamental ridge mode (in simulation or experiment), thinner DULs exhibit hybrid fundamental modes, while the thinnest DULs simply exhibit an upward shift of the fundamental mode. However, all tested chips exhibit a fundamental mode after annealing.

B. Transmission Changes in Connected Solid- and Liquid-core Waveguides

While annealing significantly improves modal confinement and loss in solid-core only waveguides, a more complete study of full optofluidic devices, that are composed of connected solid- and liquid-core waveguides, is needed. The optical transmission of combined solid-/liquid-core waveguide systems were measured over the majority of the visible light spectrum using a white light source [21]. We first examine transmission *across* a short, 12 μm wide, liquid-core waveguide. This reveals changes in the coupling efficiency across solid/liquid-core interfaces. Fig. 4a shows the improvement factor generated by dividing chip's transmission spectra before and after annealing. It is clear that the transmission of solid-/liquid-core ARROW samples improves considerably throughout the visible light spectrum. These large improvements cannot be explained by the reduced solid-core waveguide loss alone. Instead, they are due to increased transmission across the two solid-to-liquid-core waveguide intersections. This is also evident with the top-down observation that scattering at solid/liquid intersections was much brighter in as-grown samples than in annealed ones. Previous work has demonstrated the importance of ARROW intersection optimization for increased optical transmission [22]. In particular, the solid-core waveguide thickness decreases at the interface with the liquid core due to the finite conformality of the PECVD process. This crevice reduces coupling into the liquid core [22] which is much more pronounced for the DUL modes in the as-grown sample that are propagating near the top of the solid-core waveguide.

Next, we investigated transmission *through* a long (4 mm) liquid-core waveguide section. This study addresses additional changes due to multi-mode propagation in the liquid core. Fig. 4b shows a large and more spectrally varying transmission improvement due to annealing. Modeling reveals two contributions to the transmission improvement spectrum. First, the solid- to liquid-core transmission at the interface improves from 15% for the as-grown DUL sample to 96% for the annealed sample due to poor spatial overlap between the solid-core DUL and liquid-core waveguide modes. Secondly, an additional propagation effect arises from beating between the modes in the multi-mode 12 μm wide liquid core [11]. Depending on the ratio of liquid-core length and period of the beat note, spectral variations in the coupling between the solid- and liquid-core waveguides arise. This results in the observed transmission variations seen in Fig. 4b and is confirmed by simulations.

C. Repeatability of Waveguide Improvements

The effect of repeated exposure to water absorption on transmission and mode patterns of a full ARROW sensor chip with intersecting solid- and liquid-core waveguides was tested by subjecting the devices to multiple use cycles. The first point in Fig. 5 represents the transmission of an as-grown (AG) ARROW chip. The relatively low initial optical throughput is greatly improved ($> 30\times$) by the first annealing treatment (A1). In addition, the optical mode profile for an as-grown waveguide is localized in the DUL (inset), suggesting the presence of absorbed water and high refractive index that was introduced during fabrication. Conversely, the annealed waveguide shows excellent modal confinement and throughput, suggesting the elimination of water and homogenization of SiO_2 refractive index. The chip was next subjected to a typical cleaning procedure, i.e. a 4-hour boil in water (W1). The transmission decreases dramatically ($> 100\times$) upon boiling and the mode

re-localizes into the DUL due to the reabsorption of water. The annealing and water boiling processes were repeated for two more cycles, with similar changes in transmission and mode images. While transmission is always improved directly after annealing, there is a long-term decay in transmission with increasing number of annealing steps (A1–3). This is attributed to slight cracking that occurs after multiple annealing cycles, which can be eliminated by further optimization of the annealing conditions.

IV OPTOFLUIDIC SENSOR CHIP IMPROVEMENT

Finally, we address the impact of the waveguide improvement on single particle fluorescence detection in ARROW chips. For particle sensing, an orthogonal excitation/collection geometry is implemented (Fig. 6a, inset). Optical particle sensing measurements were performed similar to previously described work [9], [15]. Butt coupled laser light was used to excite analytes via a solid-core excitation waveguide (2 mW input power). This waveguide perpendicularly intersects a liquid-core ARROW in which fluorescent dye/particles were flowing (see Fig. 6a inset). Excited fluorescence signals from these analytes then propagated along the liquid-core waveguide, into the collection solid-core waveguide (through a liquid- to solid-core waveguide intersection), after which it was collected by a microscope objective, filtered by a bandpass filter, and focused onto a single-photon avalanche photodiode. Fig. 6a shows a two-fold reduction of orthogonally collected background noise upon annealing (633 nm excitation, channel filled with water). This background reduction is attributed to better light confinement within the solid-core excitation waveguide core (see Fig. 2a), which leads to less photoluminescence generation and light scattering at waveguide intersections. Next, we measured the signal intensity for bulk detection of fluorescence from 22 nM Cy5 dye solution. The detected signal shown in Fig. 6b improved by 83x for annealed chips in excellent agreement with Fig. 4 due to the improved mode coupling across the two liquid/solid-core waveguide interfaces. Combined with the background enhancement, we observe a total chip signal to noise ratio (SNR) enhancement of 166x. These improvements are fully borne out in single particle detection, seen in Fig. 6c, using 100 nm FluoSpheres ($c_F = 4 \times 10^7 \text{ mL}^{-1}$) excited by a 488 nm laser. The much improved chip performance resulted in a dramatic increase in the number of detected particles detected over a 40 second experiment from 3 particles for an as-grown ARROW chip to 314 particles after annealing. The variation of the height of the fluorescence peaks within each individual trace is due to different positions of the particles within the cross section of the fluid channel and is typical for ARROW-based single particle detection [23]. Fluorescence correlation spectroscopy (FCS) was used to determine flow velocity, and in conjunction with channel dimensions, the volume transport. Multiplying the volume transport by the concentration of particles gives an expected number of detected particles. The annealed ARROW detection chip had a 96.6 % detection efficiency, while the as-grown chip detected only 2.52 % of expected particles.

These compounded waveguide improvements have recently enabled the sensitive optical detection of single viruses [24] and even single nucleic acids [25], demonstrating the importance of thermal annealing and waveguide optimization for biosensing applications.

V CONCLUSION

We have demonstrated the presence of a high-index distinct upper layer (DUL) in PECVD grown SiO₂ ARROW ridge waveguides. This layer causes an abnormal localization of the primary waveguide mode within the DUL, leading to poor lateral light confinement, high propagation losses, and reduced mode coupling across intersections between liquid- and solid-core waveguides. Optical simulations present evidence that the DUL has higher refractive index, which is the cause of light guiding in that region.

We directly linked water absorption in SiO₂ to these degradation mechanisms in waveguide performance. Upon low temperature thermal annealing, negative effects such as poor mode localization, high propagation/intersection losses, and high background are eliminated. Furthermore, full ARROW sensor chip sensitivity is dramatically improved for detecting both bulk dye and single fluorescent nanoparticles. In summary, low temperature thermal annealing removes water from SiO₂ films, homogenizing its refractive index and consequently improves ARROW chip performance in biosensing applications. Permanent enhancement of SiO₂ waveguides can now be explored through new fabrication procedures such as deposition of reduced porosity oxides, addition of non-porous capping layers, or by creating buried channel waveguides.

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Fig. 1. Scanning electron microscope (SEM) image (left) and schematic representation (right) of a 4 μm wide solid-core ARROW ridge waveguide sitting on a silicon pedestal.

Fig. 2.

(a) Top down image of light confinement within an ARROW ridge waveguide before (top) and after annealing (bottom). (b) Integrated mode intensity output of a 1 cm long as-grown (top) and annealed (bottom) solid-core waveguide. Intensity within the core of the waveguide is shaded dark blue. (c) Transmission measurements for cut-back solid core waveguides. As-grown waveguides (open blue squares) have a fitted loss coefficient $\alpha_{AG} = 1.63 \text{ cm}^{-1}$ (dashed line). Annealed waveguides (closed red circles) have a fitted loss coefficient $\alpha_A = 0.471 \text{ cm}^{-1}$ (solid line). Both fits have $r^2 > 0.99$.

Fig. 3.

(a) SEM image of a solid-core waveguide facet with silicon substrate, ARROW layers, low refractive index SiO_2 ($n_L = 1.46$), and high refractive index SiO_2 DUL ($n_{\text{DUL}} = 1.47$) labeled as 1–4, respectively. (b) Modal simulations (top, colored) and experimental images (bottom, grayscale) for as-grown and annealed ridge waveguides. All white scale bars represent $5 \mu\text{m}$.

Fig. 4. Wavelength dependent improvement of full ARROW sensor chip transmission across (a) and through (b) the liquid-core waveguide. Insets show schematics of the transmission experiments.

Fig. 5.

Full ARROW sensor chip transmission data (see inset for experiment schematic, transmission includes two solid-core sections intersected by a liquid core channel). As-grown (AG) waveguide transmission is presented, followed by cycles of annealing (A) and 4-hour water treatments (W). Appended numbers to A and W indicate annealing/water treatment number. Mode images are alternately presented below and above the transmission data for each treatment. The axis on the right side is normalized transmission with as-grown transmission equaling 1.

Fig. 6.

(a) As-grown and annealed comparison of ARROW sensor chip performance concerning (a) orthogonally collected background (error bars are standard deviation of the background signal), (b) bulk fluorescence signal of 22 nM Cy5, (c) detection of 100 nm single FluoSpheres, $c_{\text{FI}} = 4 \times 10^7 \text{ mL}^{-1}$. The left 0.5 seconds of (c) is signal from an as-grown chip and the right 0.5 seconds is signal obtained after annealing. Representative peaks are marked with arrows. Note that all experiments were conducted with the geometry represented in the inset of (a).