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An environmental sensor based on an integrated optical whispering gallery mode disk resonator

Aaron Schweinsberg [a,*], Sandrine Hocdé [a], Nick N. Lepeshkin [a,1], Robert W. Boyd [a], Christopher Chase [b], Julie E. Fajardo [b,2]

[a] *The Institute of Optics, University of Rochester, Rochester, NY 14627, United States*
[b] *Agave BioSystems, Ithaca, NY 14850, United States*

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Abstract

In this report, we describe the fabrication and testing of an integrated optical whispering gallery mode resonator designed for use as an evanescent field environmental sensor. We use traditional photolithographic techniques to create 500 μm radius disk resonators in silicon oxy-nitride. The devices are overclad with a thin film of SiO_2 to provide a suitable surface for subsequent chemical adhesion of organic compounds and protein layers. Tests performed with sucrose solutions indicate a sensitivity to surface index variations of $\Delta n \sim 1.0 \times 10^{-5}$ refractive index units (RIU). We also demonstrate the ability of these devices to monitor protein layers binding to the surface.

Keywords: Evanescent-wave sensors; Whispering gallery mode resonators; Integrated optics; Silicon oxy-nitride; Label-free sensing

1. Introduction

Environmental sensors are an important class of devices with many applications in the food and biomedical industry including gas sensing, glucose measurement, pathogen detection, and the study of protein–protein interactions. In evanescent-wave devices, the environment is allowed to interact with the evanescent tail of an electric field mode, which then carries information about the optical properties of the environment. Sensors based on this principle offer rapid analysis and are potentially highly sensitive. The surface-plasmon-resonance (SPR), first used for immunosensing by Nylander [1], has come to be the most common commercial implementation of evanescent-wave sensors. However, there is a great deal of research into alternative evanescent-wave schemes that could provide improvements in sensitivity, robustness, device size, and ease of integration with optical sources and detectors [2,3]. Examples include integrated optical devices based on resonant mirrors, output grating couplers, Young's interferometers, and Mach-Zehnder interferometers. In principle, a resonant device, such as a whispering gallery mode (WGM) resonator, can demonstrate a significant improvement in sensitivity over Mach-Zehnder interferometers, as sensitivity is proportional to the change in transmission for a given change in the environment's index of refraction. These resonators also allow for miniaturization of the sensor, with devices containing WGM resonators millimeters or less in diameter replacing waveguides several centimeters long. Blair and Chen have proposed using the increased field strength above the surface of a disk microresonator to enhance the detection of fluorescently tagged molecules [4]. Heebner and Boyd theoretically investigated the application of microresonators to biosensing using a direct measurement of the environmental absorption and refractive index. They concluded that a detection limit of 100 molecules could be possible [5]. WGM devices have been fabricated and used as chemical sensors, with measured sensitivities to changes in the surface refractive index between 10^{-4} and 0.5×10^{-6} [6–9]. Ksendzov and Lin recently used a ring resonator waveguide in the racetrack geometry to detect avidin in solution with a sensitivity they estimate to be 0.1 nM [10]. The effectiveness of extremely high quality microspheres in sensing applications was also recently demonstrated. Vollmer et al.

* Corresponding author. Tel.: +1 585 275 5030.
E-mail address: aschwei@optics.rochester.edu (A. Schweinsberg).
[1] Present address: Department of Physics and Astronomy, San Francisco State University, 1600 Holloway Avenue, San Francisco, CA 94132, United States.
[2] Present address: Corning Incorporated, One Riverfront Plaza, Corning, NY 14831, United States.

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tracked the resonance shifts in a silica microsphere to monitor a biotin–streptavidin reaction, estimating their device to have a minimum detection weight possibly as low as 50 Da [11]. While silica microsphere WGM resonators can be made with extremely high quality factors, they are difficult to mass-produce to reliable specifications, and are not especially robust in operation, as the gap between the bus waveguide and the sphere must be mechanically controlled. We therefore chose to focus on integrated optical devices, which can be easily mass-produced once a mask is made.

In the present work, we describe the fabrication of a WGM disk resonator and show its potential for use as an environmental sensor by demonstrating its sensitivity to minute changes in the refractive index above its surface. We investigate the device's many potential applications and find it to be effective in detecting small changes of sucrose concentration in aqueous solutions, the presence of bound proteins on the surface.

2. Principle of operation

In a whispering gallery mode resonator, light is coupled from a straight waveguide, called the bus, into a disk or sphere, where the light circulates in a loop before being fed back into the initial guide. The light can be thought of as skimming around the inner surface of the resonator. These optical modes get their name from their similarity to acoustic wave propagation in the Whispering Gallery of St. Paul's Cathedral in London. The feedback of light provides the essential feature of these devices, as the returning circulating light interferes with the field that continues to enter the resonator from the bus waveguide. Whether this interference is constructive or destructive depends on the relationship between the effective path length of the whispering gallery mode and the wavelength of the light. Constructive interference occurs most strongly under the condition $N\lambda = 2\pi R n_{\text{eff}}$, where N is an integer, λ the vacuum wavelength of the light, R the radius of the resonator, and n_{eff} is the effective refractive index seen by the mode. Under this condition, light that enters the disk will circulate several times on average before reentering the bus and the device is said to be on resonance. In WGM evanescent field sensors, changes in the environment are observable as resonance shifts, since altering the refractive index of the surrounding medium changes the effective index seen by the optical mode, and, under some conditions, the effective radius of the optical path. In the case of a real device where the circulating mode experiences some loss from bending or scattering due to surface roughness, these resonance shifts can be observed by monitoring the device transmission, because light that is resonant will circulate more in the cavity, and will therefore experience more loss. Resonances correspond to dips in the transmission spectrum, the quality of the resonance being defined by the ratio of the optical frequency divided by the bandwidth of these resonances: $Q = \nu_0/\Delta\nu_{\text{FWHM}}$.

The transmission of a ring or disk resonator is given by:

$$T = \frac{a^2 - 2ra\cos\phi + r^2}{1 - 2ra\cos\phi + r^2a^2} \quad (1)$$

where r is the electric field coupling strength from the bus waveguide to the resonator, a the electric field survival factor per round trip in the resonator, and ϕ is the normalized detuning (a change in ϕ of 2π corresponds to the spacing between resonances). r is mainly determined by the spatial overlap between the waveguide mode and the WGM in the resonator. a includes contributions from scattering, bending, and material losses. Note that when $r = a$, the transmission goes to zero when the device is on resonance and this is defined to be the condition of "critical coupling." This condition is desirable for environmental sensors, as it achieves the highest possible change in transmission for a given change in ϕ.

3. Design and fabrication

Our WGM resonator devices were designed to minimize the number of steps in the fabrication process and to allow for relaxed tolerances on those steps, so that manufacture would be as rapid and inexpensive as possible. To achieve this goal, we decided to use a geometry in which the bus waveguide and resonator are laterally coupled, allowing the devices to be made with a single, reasonably deep etch. The silicon oxy-nitride material system was chosen because it allows for a wide range of refractive indices, between 1.46 (SiO_2) and 2.01 (Si_3N_4), which serves as a useful free parameter in design. Nitride films can also be grown inexpensively using plasma enhanced chemical vapor deposition (PECVD), and are compatible with silicon processing technology.

Design of the resonators was assisted by Comsol's FEMLAB. This software uses a finite element method (FEM) to solve differential equations numerically, which is helpful in estimating the coupling per unit length between the bus waveguide and the resonator. We solved the Helmholtz equation: $\nabla^2 u + (k_0 n)^2 u = \beta^2 u$ in two-dimensions over the cross-section of the ring and guide interaction region. Here u is the electric field amplitude, k_0 is the free space wavevector $2\pi/\lambda$, n the index of refraction, which is different in each material, and β is the longitudinal component of the wavevector, which can have a different value for each allowed propagation mode.

Much of the design is a trade off between different loss mechanisms, specifically between scattering loss, which occurs over any propagation, and bending loss, which attenuates light circulating within the resonator. A high effective-index contrast in the waveguide's lateral profile, attained by using a high nitride content SiON material or using a deep etch to define the guides, would reduce bending loss, but increase scattering due to surface roughness. Additionally, we wanted the bus waveguide to be single-moded at 1550 nm, which placed an upper limit on the lateral contrast. The final design incorporates 500 μm radius disks as WGM resonators, the guiding layer being a SiON film with a refractive index of 1.8 and a thickness of 350 nm. Use of a disk resonator as opposed to a ring has the advantage of decreased scattering loss, since there is only one edge from which light can scatter. However, a disk is likely to support more than one WGM, and our devices proved to be double-moded. The bus waveguides, which are used to channel light into the resonators and transmit the output are 0.8 μm wide and are evanescently

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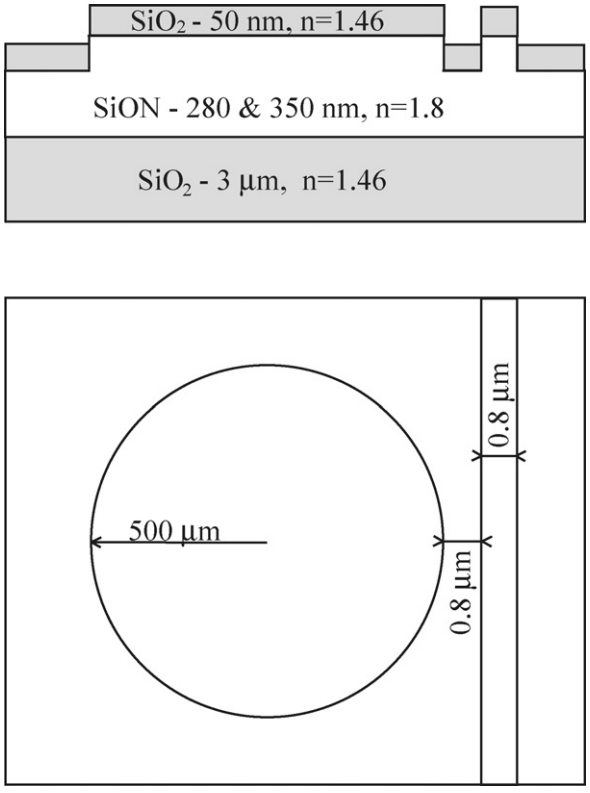

Fig. 1. Cross-section and top view of an integrated optical microdisk resonator. The waveguiding layer consists in a silicon oxy-nitride layer sandwiched by two silicon oxide layers. The thicknesses of the layers and the dimensions of the disk, gap, and waveguide are given as well as the refractive indicies of the two materials.

coupled to the disk through a 0.8 μm gap. In regions far from the resonator, the bus waveguide flares out to a width of 2.4 μm, to allow for easier coupling of light into the waveguide. Additionally, the bus waveguide contains a 90° bend so that light spilling out from imperfect input coupling can be discriminated easily from light exiting the resonator device.

We used the tools available at the Cornell NanoScale Science and Technology Facility to fabricate the resonators. A diagram of the devices is shown in Fig. 1. The first step was the growth of the vertical structure on a silicon wafer using PECVD. A 3 μm layer of SiO_2 was deposited to serve as a spacer layer between the guiding nitride film and the silicon substrate to prevent leakage of light. The nitride film subsequently deposited was measured by ellipsometry to have a refractive index of 1.8 and a thickness of 350 nm, in accordance with our design. Transverse patterning was performed using optical lithography. Fabrication of a chrome-on-quartz mask was contracted to Photronics Inc. and was performed by e-beam lithography. The waveguides were defined with a reactive ion etch (RIE) that was 70 nm deep, as verified by inspection of the edge under a scanning electron microscope (SEM). The gap width of 0.8 μm was verified with an optical microscope. In order for the surface chemistry developed for affinity biosensing to be useable with our devices, a 50 nm layer of SiO_2 was finally grown on the patterned surface using PECVD [12]. This thickness was chosen to produce an even film without appreciably diminishing the intensity of the evanescent field at the surface. Since the field of the resonator modes extends a few hundred nanometers above the surface of the nitride film, it can interact with the environment above the silica surface, either to measure bulk changes, or to detect molecules adhering to the surface itself, in the case of immunosensing.

4. Measurements and results

We characterized our finished devices using a velocity 1550 nm tunable diode laser. To access the ends of the waveguides, the chips were lapped down to a thickness of 100 μm and cleaved. They were then placed in the test setup diagrammed in Fig. 2. We used SMF-28 single mode optical fiber to couple light into the guides and collect the output. Polarization of the input light was controlled using a paddle style fiber polarization rotator. Twenty percent of the source illumination was split and recorded as a reference to correct for any fluctuations in source intensity that could occur with scanning. By imaging the top of the chips onto an InGaAs CCD camera, we were able to observe light from the resonators and waveguides while in operation. While tuned to a resonance, the transmission of the device drops sharply, in accordance with Eq. (1), as the light is held within the resonator for several round trips and experiences greater net loss due to scattering and bending of the guide. The data resulting from sweeping the laser frequency while monitoring the output intensity are shown as points in Fig. 3. It is clear from this trans-

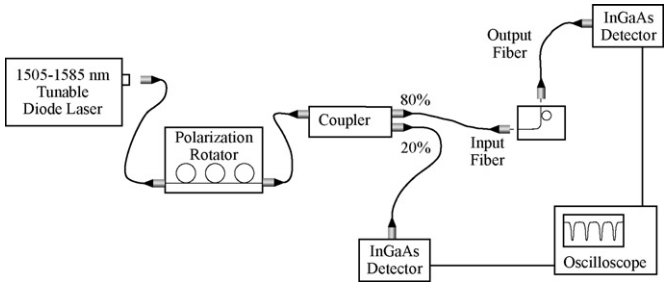

Fig. 2. Diagram of the optical test setup.

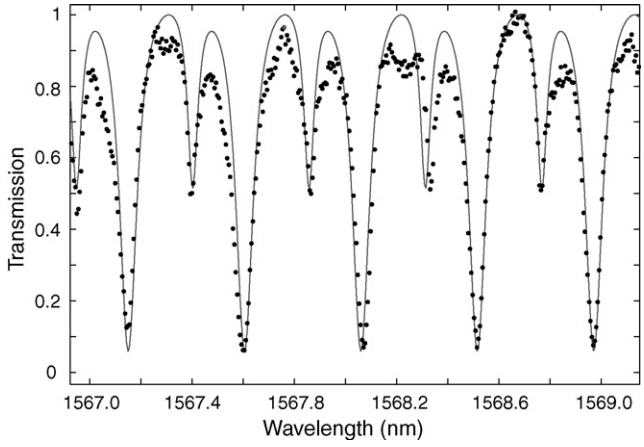

Fig. 3. Normalized transmission spectrum of a WGM resonator. The solid curve is a fit to the data model for a double-moded resonator, using a modified form of Eq. (1) and fit parameters: $a_1 = 0.7$, $r_1 = 0.8$, $a_2 = 0.75$, and $r_2 = 0.95$.

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mission spectrum that the disk supports two whispering gallery modes, the broader deep dips being associated with one mode and the narrow shallower dips with the other. Varying the input polarization reveals that only TE modes can propagate through the guides without excessive loss, so the dual resonances are clearly distinct spatial modes as opposed to separate polarization modes. We can use Eq. (1) to model our devices to extract parameters for the loss of the two modes as well as the strength of their coupling to the bus waveguide. From this simulated transmission spectrum, we estimate that: $a_1 = 0.7$, $r_1 = 0.8$, $a_2 = 0.75$, and $r_2 = 0.95$, where we define the mode indexed by 1 to be the one with deeper features. Notice that this mode is closer to the "critical coupling" condition $r = a$, for which the transmission is zero on resonance. These parameters can be used to calculate figures of merit for the WGM resonances, specifically their finesses, F, and quality factors, Q. We obtain: $F_1 = 5.7$, $Q_1 = 1.8 \times 10^4$, $F_2 = 9.4$, and $Q_2 = 3.1 \times 10^4$. The theoretical transmission spectrum for a device with such properties is shown for comparison as a solid line in Fig. 3. While this technique is adequate for performing reasonably sensitive detection of surface changes, we note that losses are somewhat higher than expected and that significant improvements in performance over these parameters should be possible with modifications to the fabrication process.

To test the resonators' performance as environmental sensors, we monitored changes in the transmission spectrum with very small changes of the surface refractive index. Frequency modulation of the diode laser output was used to follow a single narrow resonance over a 0.45 nm wide spectral range. We locked in on a single device resonance and followed it as the refractive index of the surface layer was modified. A representative set of data for this type of experiment is shown in Fig. 4. In this experiment, a drop of distilled water was initially placed on the disk. Trace 1 shows the position of this reference resonance. A droplet of an aqueous sucrose solution at 5.3 mg/ml was then delivered via pipette to the water on the surface. Trace 2 shows that this addition caused the resonance to shift higher in wavelength by 0.1085 nm. After adding another drop of this solution, the resonance was observed to move in the same direction by an additional 0.084 nm, as shown in Trace 3. All liquids used in the experiment were at room temperature. We estimate a resolution of 0.005 nm at the point of maximum slope on the resonance, and from this, can calculate the disk resonator's sensitivity. Multiple measurements of the resonance shift give values for the minimum detectable surface index change between 0.5×10^{-5} and 1.5×10^{-5} RIU, clustering around 1.0×10^{-5}. The large spread of values is likely due to small deviations in temperature, possibly including the effects of evaporation, causing the actual index of the sucrose solution to differ slightly from the literature value at room temperature. In order to obtain a precise value for the concentration of a sucrose solution, and not merely its refractive index, one would need to use the resonator sensor in a setup where the temperature of the liquid could be rigorously controlled.

We performed further experiments to test the applicability of the WGM devices to immunosensing. The goal was to observe real-time changes in the transmission spectrum as proteins attach to the device's surface. In order to do this it was necessary to develop a method for consistent immobilization of proteins to the surface of the SiO_2 coated devices. Since epoxides are highly reactive to amines, we decided to use an epoxy silane as an intermediary to enable attachment of antibodies to the silica surface. Upon exposure to amines the epoxide ring breaks open and a covalent linkage is formed between the amine of the protein and the silane, which has been previously bound to the surface (Fig. 5). Epoxides also react less strongly with alcohols and only weakly with carboxylic acids and other epoxides. For deposition of the epoxy silane layer, the silica coated chips are carefully cleaned. A well-stirred 2% epoxy silane ((3-glycidoxy)trimethoxysilane) mixture in 95% ethanol is then poured onto the clean chips and left 2 min at room temperature. The epoxide coated chips are rinsed to remove residual silane, and baked for 15 min at 110 °C to covalently attach the silane to the silica. By using a microarray laser scanner to examine fluorescently tagged primary and secondary antibodies washed onto the chips, we were able to verify that the above procedure was successful in producing a surface that enabled covalent attachment of proteins.

Using a solution of mouse anti-goat IgG antibodies, we performed an experiment to detect protein adhesion to the surface of the WGM resonator. An epoxide coated chip containing the

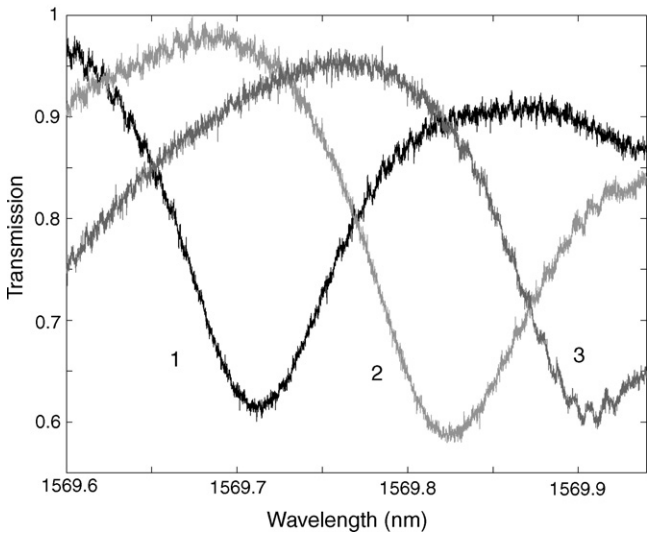

Fig. 4. Resonance shift of a 500 μm radius disk as its surface layer is modified. Trace 1 represents the initial resonance with a droplet of distilled water covering the disk. Two drops of a diluted aqueous sugar solution were added successively to the water droplet (Traces 2 and 3).

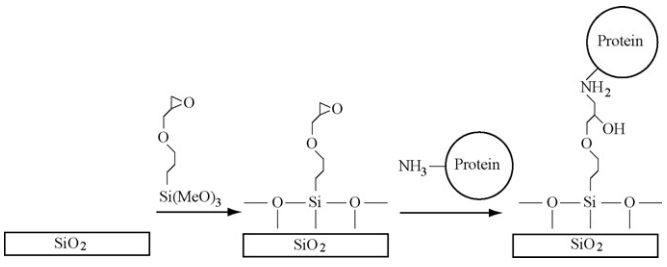

Fig. 5. (3-Glycidoxypropyl)trimethoxysilane deposition on silica and subsequent protein binding to the epoxide.

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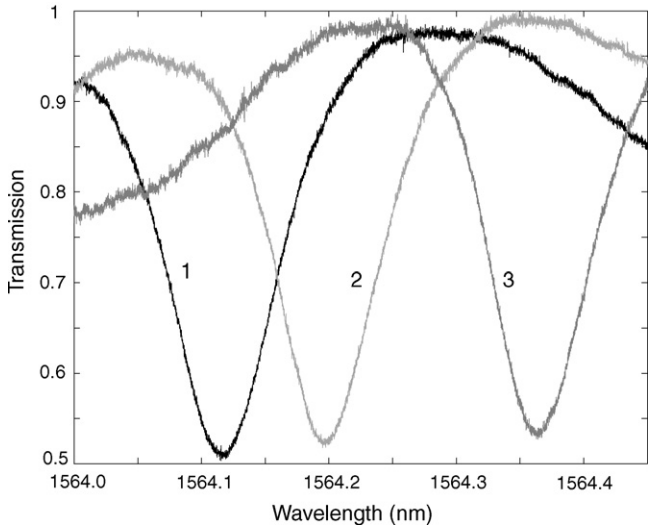

Fig. 6. Shift of WGMR resonance with deposition of antibodies on the surface. Trace 1 shows the initial resonance after the 1×PBS was placed over the waveguide surface. Trace 2 represents the system after the addition of 0.18 mg/mL IgG solution to the initial drop. Trace 3 shows the evolution of the system after 4 min.

device was placed in the optical test setup and aligned to the 1550 nm tunable source. After depositing a 0.6 μL drop of phosphate buffered saline (1×PBS) on the disk, we focused on a particular resonance. We then mixed a solution of mouse anti-goat IgG in PBS at a concentration of 0.18 mg/mL and used a pipette to apply 0.6 μL of this solution on top of the first drop. A rapid shift in the resonance position of 0.081 nm immediately followed. Over time, the resonance was then observed to shift further, acquiring an additional displacement 0.167 nm over a period of 4 min, which we believe is due to the slow accumulation of additional proteins on the surface. These results are shown in Fig. 6.

In another experiment, designed to serve as a control, we took a chip coated with epoxide and placed on it a 0.6 μL drop of PBS. We then added a 0.6 μL drop of IgG solution at 1.8 mg/mL concentration, and observed a large shift of the resonance position of about 0.5 nm. We then washed the surface of the chip three times with diluted buffer and ultrapure water. After adding another drop of buffer, a second drop of the IgG solution only caused a slight displacement of the resonance by 0.03 nm, indicating that most of the wavelength shift in the first experiment comes from antibodies on the surface, as opposed to proteins in suspension.

It is worth noting that while we have chosen to use a tunable source to perform measurements, it is not the only mode of operation for these devices. It is also possible to track changes in the resonance position by using a broadband source, such as an LED or fluorescent emission from an erbium-doped fiber, and a sensitive spectrometer at the output. Also it is possible to use a simple monochromatic source and photodiode detector, but it would be necessary to implement some form of feedback at the chip level if it were necessary to maintain operation in the spectral region of highest sensitivity. One system for performing such feedback in nitride evanescent field sensors was described by Heideman and Lambeck [3].

5. Conclusion

In summary, we have developed environmental sensors based on laterally waveguide-coupled whispering gallery mode resonators in silicon oxy-nitride. The design is suitable for mass production as they were fabricated using the widely known processing technologies of PECVD, optical lithography, and RIE. The highest quality resonators have a Q of approximately 3.1×10^4 and a finesse of 9.4. Experiments with sucrose solution demonstrate a sensitivity to surface refractive index changes as small as 1.0×10^{-5} RIU. We have also shown that the devices can monitor the attachment of a protein monolayer to the surface. These experiments demonstrate the potential of our sensors for immunosensing or the monitoring of protein–protein interactions in general. We believe that further improvements in fabrication procedure could potentially reduce device losses and increase the quality of the resonator, improving device sensitivity considerably.

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Biographies

Aaron Schweinsberg was born in Lewisburg, Pennsylvania. He received a BS degree in applied and engineering physics from Cornell University in 2000. He is currently pursuing a PhD in optics at the University of Rochester under the supervision of Dr. Robert Boyd. His research interests include quantum coherence effects in semiconductor heterostructures, slow and fast light phenomena, nonlinear optical effects in composite materials and optical waveguides, and environmental sensing with waveguides. He is a member of the American Physical Society.

Sandrine Hocdé received her PhD in materials science in 2000 from the University of Rennes, France. Before working as a glass and fiber engineer for NP Photonics, Inc., in Tuscon, Arizona, she was a research associate at the Optical Sciences Center at the University of Arizona; she joined the University of Rochester and worked at the Institute of Optics. She is currently conducting her research in the Department of Biomedical Engineering. Her research interests are in the fields of glass, optical fibers, evanescent wave spectroscopy, optical biosensors, and total internal reflection fluorescence microscopy.

Nick N. Lepeshkin was born in Dudunka, Russia, in 1974. He received his PhD in physics from New Mexico State University in 2001. He then spent several years doing postdoctoral research work at the Institute of Optics, University of Rochester. He is currently an assistant professor in the Department of Physics and Astronomy at San Francisco State University. His research interests are in optical physics, nonlinear optics, and integrated optical devices.

Robert W. Boyd was born in Buffalo, New York. He received the BS degree in physics from the Massachusetts Institute of Technology and the PhD degree in physics in 1977 from the University of California at Berkeley. His PhD thesis was supervised by Professor Charles H. Townes and involves the use of nonlinear optical techniques in infrared detection for astronomy. Professor Boyd joined the faculty of the Institute of Optics of the University of Rochester in 1977 and since 1987 has held the position of professor of optics. Since July 2001 he has also held the position of the M. Parker Givens professor of optics, and since July 2002 has also held the position of professor of physics. His research interests include studies of "slow" and "fast" light propagation, quantum imaging techniques, nonlinear optical interactions, studies of the nonlinear optical properties of materials, the development of photonic devices including photonic biosensors, and studies of the quantum statistical properties of nonlinear optical interactions. Professor Boyd has written 2 books, co-edited 2 anthologies, published over 230 research papers, and been awarded 5 patents. He is a fellow of the Physical Society and the Optical Society of America and is a past chair of the Division of Laser Science of the American Physical Society.

Christopher Chase is a nanofabrication specialist at Agave BioSystems. He received an AAS degree from Broome Community College in 1988 in the field of chemical engineering technology. Mr. Chase works on the fabrication and characterization of micro- and nanoscale fabricated devices and microfluidic structures. He was awarded a patent on a method for detection of biomolecular interactions, and has co-authored several scientific papers. He is also responsible for characterizing various microorganisms and structures and for the development of photolithographic techniques and methods for Agave BioSystems.

Julie E. Fajardo is a staff scientist at Agave BioSystems. Ms. Fajardo received an MS and BS from Rutgers University in New Brunswick, NJ, in the Department of Microbiology and Molecular Genetics and the Department of Chemical and Biochemical Engineering, respectively. At Agave BioSystems, Ms. Fajardo uses a combination of molecular biology and biochemistry techniques for the development of biosensors. She is currently the principal investigator on a project to identify and isolate shikimate pathway genes from the parasite *Plasmodium falciparum* for applications in malarial drug discovery. In recent collaborations with Cornell University and the University of Rochester, Ms. Fajardo has led projects on the development of electrochemical and optical detection systems for DNA hybridization and protein–protein interactions. Ms. Fajardo is also involved in the development of quantum dot dipstick assays for the detection of the parasite *Leishmania*.

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