Development of Silicon-Based Optofluidic Sensors for Oil Sands Environmental Monitoring

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Oil Sands Research and Information Network

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REPORT SUMMARY

The oil sands industry in Alberta produces large volumes of process-affected water (PAW), which is known to contain heavy metals and organic compounds (such as naphthenic acids, naphthalene, phenanthrene, pyrene, etc.) that are toxic and hazardous to the environment. The industry has an ongoing need to improve the monitoring of concentrations and breakdown of these compounds. Currently, this is mainly accomplished by collecting samples for shipment to a laboratory for analysis. Portable and ideally distributed and real-time monitoring techniques would greatly improve efficiency and the base of knowledge with respect to these environmental concerns.

The principal aim of the project was to develop a prototype lab-on-a-chip (LOC) based sensor for optical detection of target molecules in PAW using spectrally resolved fluorescence detection. The proposed sensor would offer a high level of integration between the fluidic and optical components, potentially reducing the cost and complexity of the overall system while also improving the performance (sensitivity, signal to noise ratio (SNR), alignment tolerance, etc.). In the long term, such miniaturized sensors hold promise as low-cost, highly distributed environmental monitoring devices.

Most of the primary milestones of the project were successfully completed, as follows:

1. A silicon-based air-core waveguide technology was developed and optimized for the ultraviolet-visible wavelength band of interest. These waveguides employ low-loss TiO_2/SiO_2 Bragg reflectors deposited by sputtering deposition at the U of A nanoFab.
2. Tapered air-core waveguides were assembled and tested as visible-band micro-spectrometers. These micro-spectrometers provide resolution on the order of 1 nm over a 100 nm operational band (e.g., wavelengths in the 500 to 600 nm range), and offer compelling advantages for lab-on-a-chip and optofluidic microsystems.
3. Prototype sensing systems were developed, by combining the aforementioned micro-spectrometers with PDMS-based microfluidics. Fluorescence spectroscopy was successfully demonstrated for commercial dyes with fluorescence bands in the ~500 to 600 nm wavelength range.

At the time of writing, ongoing work is aimed at translating the operational band of these sensors to the ~400 to 500 nm wavelength range. This effort has been delayed by processing difficulties, but is expected to reach a successful conclusion in summer 2013. Further work is aimed at extending the operational range of the micro-spectrometers (e.g., 400 to 650 nm), by using more sophisticated multilayer designs. We hope that this work will enable the detection of native fluorescence from hydrocarbon molecules, including the multiplexed detection of multiple species, and intend to pursue this objective in the coming months.

ACKNOWLEDGEMENTS

The Oil Sands Research and Information Network (OSRIN), School of Energy and the Environment (SEE), University of Alberta and the Canada School of Energy and Environment Ltd. provided funding for this project.

1 INTRODUCTION

Oil sands tailings ponds present significant environmental and reclamation challenges. In particular, naphthenic acids as a byproduct of oil sands refining represent a significant hazard to water systems and animals (Allen 2008, Rogers et al. 2002). Currently, monitoring of these contaminants relies mainly on the collection of samples from tailings ponds and transport to remote laboratories equipped for detailed sample analysis[1]. Portable analysis equipment based on optical fluorescence detection have been proposed and implemented (Kavanagh et al. 2009, Taschuk et al. 2013), however using bulk optical componentry and commercial spectrometers. Such schemes are still relatively bulky and expensive, and require significant power. Nevertheless, optical sensors have well-known advantages, including immunity to electromagnetic interference, compatibility with harsh environments, and potential for non-destructive analysis of samples. Miniaturization of sensors using microfluidics and lab-on-a-chip (LOC) techniques is a long-held goal and there is great interest in chip-scale optical sensors, enabled by co-integration of microfluidics and integrated optics. However, the development of LOC sensors is currently hindered by the lack of integration of optical detection methods. To date, most systems rely on large-scale and expensive off-chip optical devices such as confocal microscopes and optical spectrum analyzers (Meyers and Lee 2008). For applications such as field-deployable toxin and environmental hazard detectors, this has delayed the widespread commercial deployment of LOC techniques.

The main objective of this project was the development of a portable sensor technology for the detection and identification of hydrocarbon molecules (based on their native fluorescence spectra) in process-affected waters (PAW) produced by the oil sands industry. The hypothesis was essentially that we could apply a previously developed set of technologies (integrated hollow waveguides and micro-spectrometers based on tapered hollow waveguides) to the implementation of chip-scale systems that can enable laser-induced fluorescence (LIF) spectroscopy of liquid-phase samples. We proposed that this chip-based technology (with optical part < 1 cm^2) could replace the functionality provided by existing systems based on bulk optics and commercial spectrometers, which are relatively expensive and much larger in size.

Our methodology was as follows:

1. We developed a micro-spectrometer operating in a suitable range (UV-visible region) for UV-induced fluorescence of unlabeled hydrocarbon molecules.
2. We integrated this micro-spectrometer technology into a prototype optofluidic system to facilitate the efficient excitation of a small-volume liquid sample using a laser or LED source, and the efficient collection of fluorescence light by the micro-

[1] See Zhao, B., R. Currie and H. Mian, 2012. Catalogue of Analytical Methods for Naphthenic Acids Related to Oil Sands Operations. OSRIN Report No. TR-21. 65 pp. http://hdl.handle.net/10402/era.26792 for information on traditional lab-based analysis of naphthenic acids.

spectrometer part. Various strategies involving PDMS-based microfluidics or micro-capillaries were explored.

3. Preliminary proof-of-concept testing of the prototype sensors employed commercial fluorescent dyes to confirm the basic operation of the micro-system. These dyes were selected based on the operational range (~500 to 600 nm wavelength) for the micro-spectrometers fabricated to date.

4. In ongoing work, we are attempting to detect and resolve native fluorescence from hydrocarbon samples. Typically, these molecules fluoresce in the ~350 to 500 nm wavelength range, outside the range supported by the first-generation prototype mentioned above. Second-generation samples tuned to this band are under development.

2 RESULTS

We aimed to implement a chip-scale, spectrally resolved fluorescence detection sensor, in which all components except for the light source are integrated onto a single chip. The proposed system would offer close integration between microfluidic and optical componentry, and builds on previous work in our laboratory:

1. We developed a novel hollow waveguide and microcavity technology using silicon-compatible materials (Allen et al. 2011, Epp et al. 2010). These hollow waveguides are capable of acting as both an optical waveguide and a microfluidic channel, so that light-analyte interactions can be significantly enhanced compared to conventional approaches.

2. We developed a miniature micro-spectrometer based on tapered versions of our hollow waveguides (DeCorby et al. 2009), as illustrated schematically in Fig. 1.

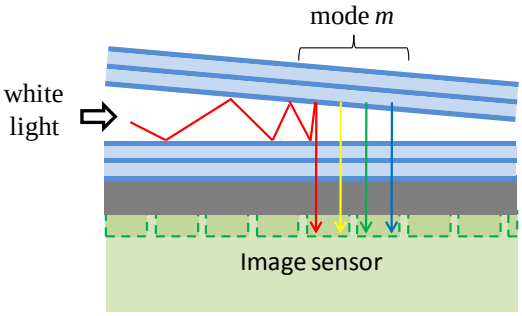

Figure 1. Schematic illustration of a tapered waveguide spectrometer. The waveguide comprises an air core (cavity) bounded by omnidirectional Bragg reflector mirrors. Polychromatic light is spatially dispersed due to the spectral dependence of the mode cutoff position in the taper. Due to the omnidirectional reflection properties of the claddings, the cutoff light is vertically radiated from the waveguide at cutoff and the spatial pattern can be captured by an image sensor. Spatial-to-spectral mapping of this pattern is the basis of a compact spectrometer.

The goal of the project was to adapt and optimize these technologies for the on-chip detection of native fluorescence from hydrocarbon molecules in process-affected water. To this end, we developed new waveguide mirrors, new techniques for tapered waveguide assembly, and new strategies for optofluidic integration.

The one-year project encompassed four milestones, and progress towards each of these milestones is described in the following sub-sections. With respect to the milestones, the project has proven to be a nearly complete success. There is admittedly some optimization work that remains, however, especially related to packaging and fluidic interface to the prototype sensor.

2.1 Milestone 1: Studies on Fluid Flow Through Hollow Waveguides

We conducted an extensive series of experiments on interfacing external fluid connectors to our hollow waveguide chips. These mainly involved the use of PDMS (a rubbery polymer employed extensively in microfluidics). Two different strategies were explored:

1. We deposited PDMS layers directly onto spectrometer chips containing channel waveguides, and developed a technique to connect micro-fluidic syringe interfaces to these chips. Full details will be described in Brian Drobot's M.Sc. thesis (Drobot 2013). This development was not completely successful, as we discovered it was very challenging to pump fluids through our channel waveguides, due mostly to their small cross-section (they are a few μm tall, typically). Furthermore, we anticipated significant challenges with respect to real-world PAW samples (due to particulates).
2. These challenges prompted us to explore alternative strategies, in which the hollow waveguide based spectrometer is simply placed in close proximity to a separate microfluidic channel or reservoir (such as a micro-capillary or PDMS-based channel), as shown for example in Fig. 2. This strategy led to new insights into superior ways for applying our spectrometer to fluorescence spectroscopy, which is the subject of a Report of Invention filed with Tec Edmonton in late 2012 and described in greater detail below.

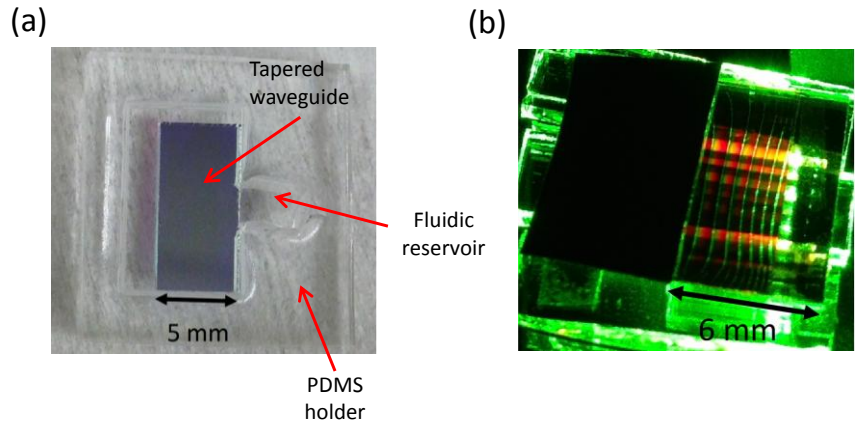

Figure 2. Photographs of taper spectrometer chips interfaced to PDMS microfluidics. (a) the photograph shows a taper spectrometer chip placed in a pre-fabricated PDMS holder, (b) The photograph shows light collected and radiated by a taper; approximately 7 modes are present in the radiated light. The green lines are the cutoff radiation associated with the green laser light used to excite a laser dye. The bands of orange and red are the dispersed light from a fluorescent laser dye.

2.2 Milestone 2: Development of Hollow Waveguides for Visible Wavelengths

We adapted our (recently patented) technology for fabrication of on-chip, air-core channel waveguides using stress-driven buckling assembly. This work involved the development and optimization of thin film deposition processes at the U of A nanoFab[2], using SiO_2 and TiO_2 as low and high index mirror layers (see Fig. 3). Significant progress was made, but the process proved to be somewhat sub-optimal due to the limited amount of compressive stress we were able to realize using the available sputtering system.

As a result of the challenges mentioned above, we decided to employ a back-up strategy for implementation of the proposed spectroscopic sensors. A hybrid integration technique (see Fig. 4) based on wafer bonding was used to fabricate hollow slab waveguides. By using appropriate mirrors, we are able (in principle) to customize the spectrometer operating range to lie within any part of the visible spectrum (with a typical operating bandwidth of ~100 nm).

[2] See http://www.nanofab.ualberta.ca/

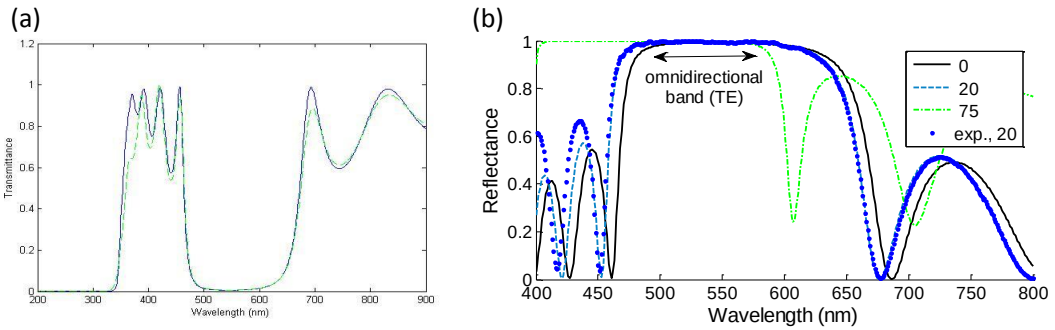

Figure 3. Results from the development of sputtered SiO_2/TiO_2-based Bragg mirrors and tapered waveguides are shown.
(a) Normal-incidence transmittance for a typical mirror is shown, with the stop-band spanning the wavelength range ~480 to 650 nm. The solid curve shows theoretical predictions and the dashed curve is the experimental result.
(b) Reflectance versus wavelength for several incidence angles and for TE polarization. The operating range of the spectrometer is defined by the omnidirectional band of the mirrors, in the case shown spanning the wavelength range ~490 to 570 nm.

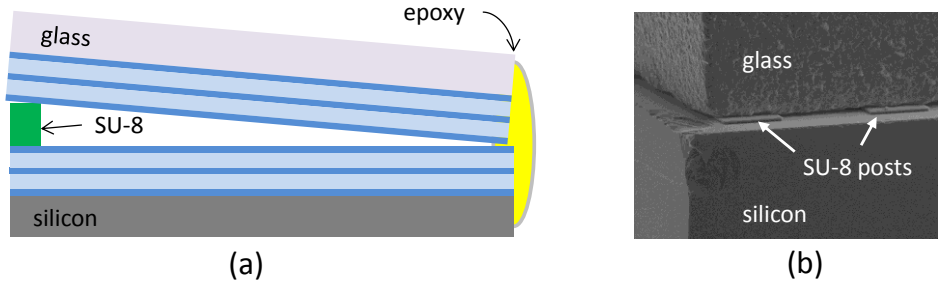

Figure 4. A tapered slab Bragg waveguide.

(a) Schematic illustration of a tapered slab Bragg waveguide fabricated by bonding a glass substrate to a patterned Si substrate.
(b) SEM image showing the end facet (wide end) of a taper. For scale reference, the spacing between adjacent SU-8 posts is 150 μm.

In addition to providing simplified assembly and fabrication, the slab waveguide tapers provide additional advantages for the fluorescence spectroscopy application of interest:

1. The input aperture is effectively much larger, since the slab waveguide can be arbitrarily large in the transverse direction (i.e., normal to the page in Fig. 4(a)). This increases the light collection efficiency of the spectrometer.

2. The taper height and slope can easily by varied by adjusting the height of the SU-8 posts, allowing the spectral dispersion and number of supported modes to be customized.

Details of the fabrication process can be found in a recent publication (Drobot et al. 2012).

2.3 Milestone 3: Development of Visible Band Waveguide Spectrometer

Using the hybrid assembly technique, we were able to implement waveguide tapers with air-core height tapered from approximately 2 to 5 μm (i.e., at the wide end of the taper) to approximately 0 μm over a distance of several millimetres. These tapers were coupled (via suitable optics) to a CMOS or CCD-based image sensor. Resolution on the order of ~1 to 2 nm, with an operating bandwidth of ~100 nm in the visible region was verified, and full details can be found elsewhere (Drobot et al. 2012). As an example, Fig. 5 shows the spectral dispersion of an input white light source. Each 'rainbow' band corresponds to the light associated with cutoff of a particular waveguide mode.

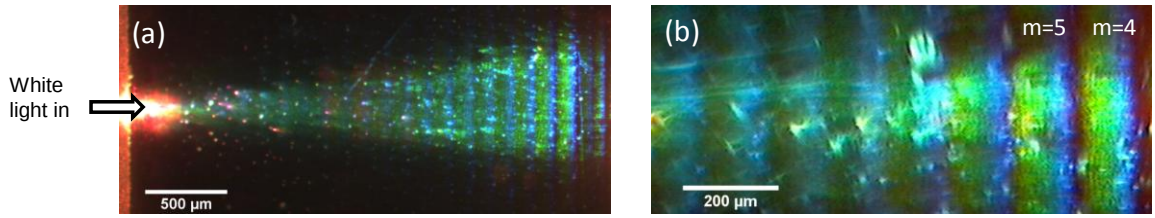

Figure 5. Spectral dispersion of visible-band white light by a tapered air-core Bragg waveguide.
The camera captures the spectrum of the input light from the spectral dispersion of the cutoff point for each mode order (m).
After Drobot et al. (2012).

2.4 Milestone 4: Assembly and Test of Prototype Sensing System

A schematic of the prototype sensing system is shown in Fig. 6. Several variations on this design have been conceived, and are described in greater detail in DeCorby (2012). In work to date, two different strategies have been pursued for interfacing fluid samples to the micro-spectrometer. In the first approach, tapered waveguide chips were embedded into custom-built PDMS microfluidic holders (see Fig. 2 above), so that fluid samples could be introduced near the wide end of the tapered waveguide. In the second approach, fluid samples were sealed in small sections of commercial micro-capillaries. These capillaries were then simply assembled in close proximity to the waveguide tapers, so that fluorescence light could be coupled with reasonable efficiency. Further details can be found elsewhere (Drobot et al. 2013).

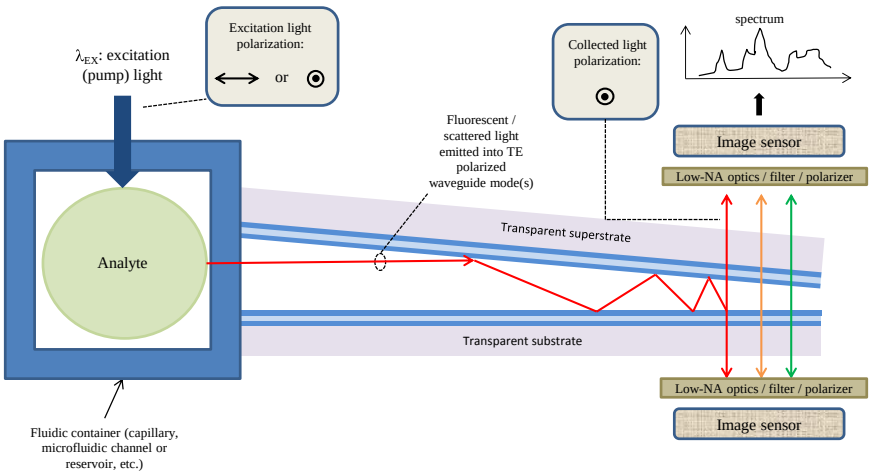

Figure 6. A general schematic of the proposed micro-system for spectrally resolved detection of the light scattered or fluoresced by an analyte.
Some of the fluorescent or scattered light from the analyte is coupled into resonant waveguide modes, and propagates towards the narrow end of the taper. Owing to the polarization-dependent properties of the cladding mirrors (for the case shown), only TE-polarized modes propagate with low loss, and the waveguide acts as a very efficient polarizer. A significant fraction (typically ~0.1) of the light in the low-loss TE-polarized modes is spatially dispersed and radiated in a nearly vertical direction at mode cut-off. This radiated light can be detected by an image sensor (photodetector array), placed either above or below the tapered waveguide, and used to reconstruct the spectrum of the fluorescent or scattered light.

As mentioned above, first-generation prototypes operate in the ~500 to 600 nm wavelength range. For proof-of-principle testing purposes, we obtained commercially available fluorescent dyes (Fluospheres, Invitrogen Inc.) with emission spectra matched to this operational range. Figs. 7 and 8 show example spectra extracted from experiments using these commercial dyes (Drobot et al. 2013).

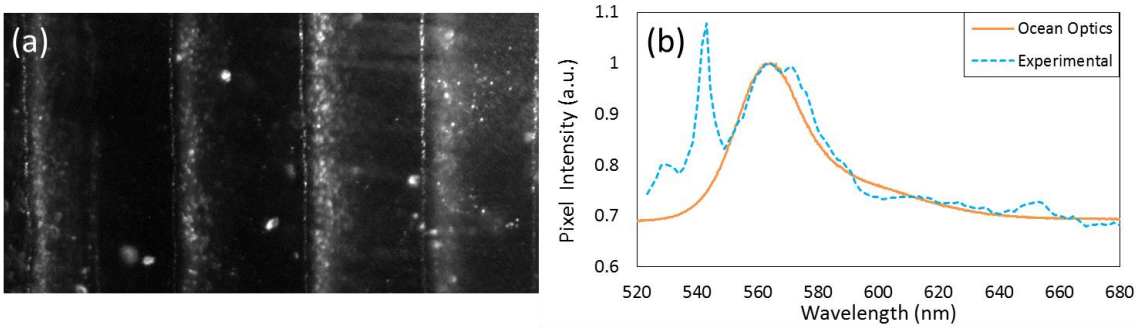

Figure 7. Fluorescence spectrum of Orange FluoSpheres.
(a) Camera image of spectrally-resolved fluorescence spectra from 'Orange' FluoSpheres (Invitrogen, Inc.) excited near the wide end of a taper. Mode orders m = 2 to m = 5 are shown. The discrete lines are due to scattered light from the 543 nm laser used to excite the fluorescence.
(b) Fluorescence spectrum of the Orange FluoSpheres (and the residual laser light at 543 nm), extracted from the radiated light collected from mode order m = 2. As shown, the fluorescence spectrum agrees well with that collected using a commercial spectrometer.

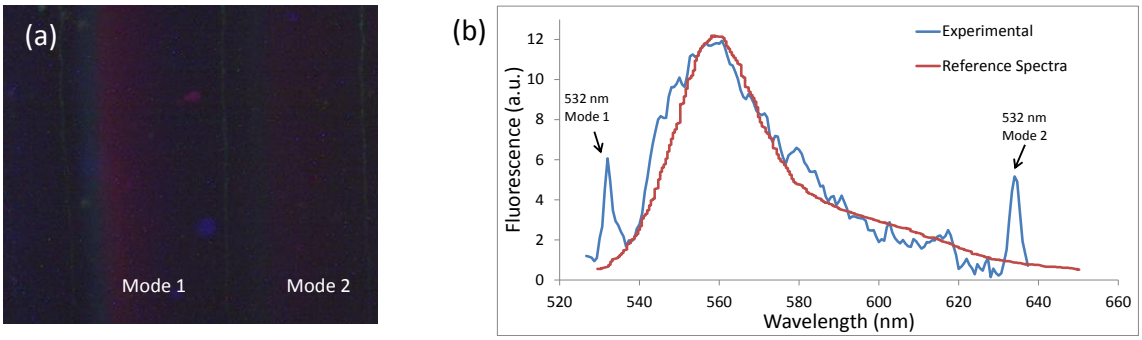

Figure 8. Fluorescence spectrum of Orange FluoSpheres using a 532 nm wavelength excitation laser.
(a) Camera image of spectrally-dispersed light associated with cutoff of mode orders 1 and 2.
(b) Fluorescence spectrum extracted from mode order 1.

While these results confirmed the general principle, we believe that significant optimization is possible by using improved taper design, optics, and microfluidic interface (Drobot et al. 2013).

3 ONGOING AND FUTURE WORK

3.1 Ongoing Work

The primary piece of unfinished work is the spectral detection of native (UV) fluorescence from target hydrocarbon samples. We fully intend to pursue this goal, and hope to achieve the milestone during the summer of 2013. Key personnel (M.Sc. student Aaron Melnyk, summer student Min Choi) are still working in our lab, and will be leading the hands-on portion of the work. In addition to the work described above, we have made the following progress towards this goal:

1. Through a collaboration with the Tsui group (Taschuk et al. 2013), we have obtained hydrocarbon samples and verified their fluorescence spectra using conventional spectrometry.
2. We have successfully developed Bragg mirrors for the target range of interest near 400 nm wavelength (see Fig. 9).

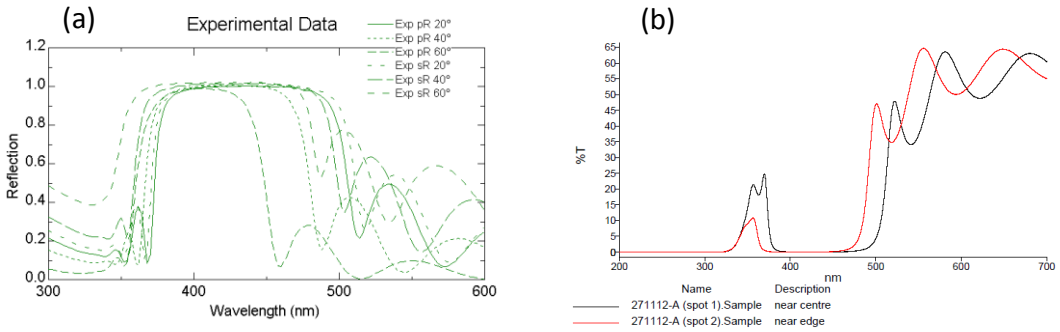

Figure 9. Experimental results for Bragg mirrors centred near 400 nm wavelength.
(a) Reflectance versus wavelength for various incident angles
(b) Normal-incidence transmittance.

Completion of this milestone has been delayed by unexpected problems with tapered waveguide assembly. These problems are mundane in nature, arising from challenges with respect to the definition of the SU-8 features (see Fig. 4). We don't anticipate any fundamental barriers to final prototype implementation, once these processing issues are sorted out.

3.2 Future Work

There are several interesting strategies to explore in future work. For example, the main limitation of the existing prototype is its operational range, since the micro-spectrometer chips we have realized to date can resolve spectral content over ~100 nm of the wavelength spectrum in the visible range. This is already very useful, especially if the part is specifically designed to target a specific molecule with native fluorescence centred at a known wavelength. However, it would be preferable if the operational range of the sensor could be expanded, for example to

cover the entire 400 to 700 nm wavelength range of the visible spectrum. This would allow flexibility in terms of identifying multiple target molecules (possibly simultaneously) with a single chip. There are at least two promising strategies to achieve this goal, as follows:

1. We aim to explore the use of higher index contrast mirror materials. For example, GaP is an interesting high-index material (Gao et al. 2011). Alternatively, photonic heterostructures (multilayers containing two or more offset quarter-wave-stacks) could be explored.
2. We believe that a single PDMS enclosure could easily be designed to hold multiple taper spectrometer chips, each independently customized to cover a unique part of the spectrum. For example, three taper chips with their omnidirectional stop-bands offset by 100 nm will allow us to design a system that operates over the entire visible spectrum (~400 to 700 nm wavelength) using the materials employed to date.

A major emerging theme within LOC-based sensors is the use of cell-phones to support POC and distributed instrumentation. This is partly motivated by the incredible market penetration of cell-phones (there are estimated to be ~6 billion cell-phones in use, most in the developing world). The effort is furthermore supported by the impressive computing power, convenient software development interface, and optoelectronic capabilities (i.e., the CMOS-based image sensor) embedded within these devices. Many research groups, especially the Ozcan group at UCLA (Zhu et al. 2011) have already demonstrated impressive results using cell-phones as a basis for low-cost, portable microscopy, with applications to diagnostics and sensing.

To date, there have only been a few efforts to implement a spectrometer using a cell-phone platform. Smith et al. (2011) combined a commercially available diffraction grating with a cell-phone camera, and achieved a reasonably good resolution (~10 nm) in the visible part of the spectrum. However, the device required the attachment of a collimating tube (~10 cm in length) with an entrance slit, to ensure only collimated light reached the diffraction grating. It is not clear that such a device would have great utility in the context of LOC-based POC diagnostics. We believe that we have potentially developed a better solution for such applications. In future work, we hope to develop a high-resolution spectrometer attachment for cell-phone-based diagnostics and sensing (see Fig. 10).

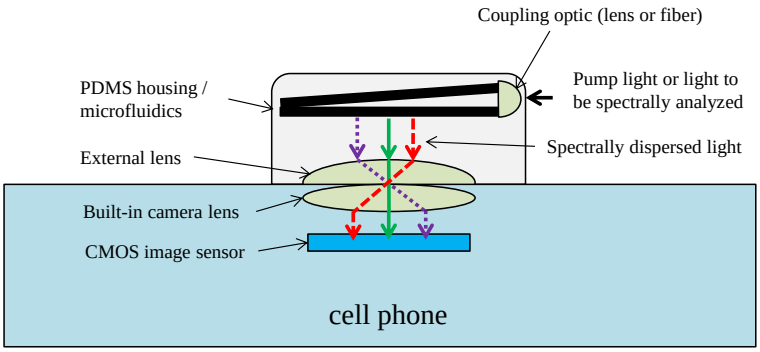

Figure 10. Proposed future implementation of the LOC spectral sensor as field-deployable and portable optofluidic system.
The optical and microfluidic parts will be designed to clip or mount onto the camera lens of a standard smart phone. The smart phone will provide the image capture and data analysis to facilitate quantitative analysis of target molecules.

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5 GLOSSARY

5.1 Terms

Bragg Mirror

An optical mirror based on two or more dielectric materials, where reflection from multiple interfaces is made to constructively interfere so as to produce high reflection over a certain range of wavelengths.

Diffraction Grating

An optical device with periodic apertures that produces a spatial dispersion of polychromatic light.

Fluorescence

Light emitted by a molecule following absorption of shorter-wavelength light.

Lab-on-a-chip

Micro-systems that incorporate microfluidic channels and related elements to facilitate sensing, chemical reactions, etc. on a chip.

Microcavity

A micro-scale optical resonant cavity.

Optofluidics

Micro-systems that combine microfluidics and integrated optics, often for sensing or detection in lab-on-a-chip systems.

Spectrally-Resolved Fluorescence Detection

The detection of the range of wavelengths emitted by a fluorescent molecule, as opposed to simply detecting the emitted power, for example.

Spectrometer

An optical instrument that provides the spectral content of an input optical signal.

Sputtering / Sputtered

A thin-film deposition technique based on the acceleration of noble gas ions towards a target.

Waveguide

A structure that confines light (or electromagnetic energy, in general) to propagate along a given direction.

5.2 Acronyms

CCD	Charge-coupled device
CMOS	Complementary metal-oxide-semiconductor

LED	Light Emitting Diode
LIF	Laser-induced Fluorescence
LOC	Lab-on-a-chip
OSRIN	Oil Sands Research and Information Network
PAW	Process-affected water
POC	Point-of-care
SEE	School of Energy and the Environment
SEM	Scanning electron microscope
SNR	Signal-to-noise-ratio
TE	Transverse electric
TM	Transverse magnetic
UV	Ultraviolet

5.3 Chemicals

GaP	Gallium phosphide
PDMS	Polydimethylsiloxane
Si	Silicon
SiO_2	Silicon dioxide
TiO_2	Titanium dioxide

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