Integrated planar Bragg grating oxygen sensor

D.J. Wales, R.M. Parker, J.C. Gates, M.C. Grossel, P.G.R. Smith

Optoelectronics Research Centre & School of Chemistry
University of Southampton
Southampton, U.K.
dw405@soton.ac.uk

Abstract— **We demonstrate an integrated planar Bragg grating sensor for the detection of oxygen by modification of the surface with a silica sol–gel containing immobilized methylene blue.**

Keywords: oxygen sensing; Bragg gratings; evanescent field; methylene blue; silica sol–gel.

I. INTRODUCTION

The detection of oxygen is required in a diverse set of scientific fields. We shall present a planar integrated Bragg grating sensor with a chemically modified overclad for the detection of oxygen in gaseous or liquid environments.

A. Oxygen sensing

Quantitative and qualitative oxygen sensing is required for the understanding of many chemical processes across a diverse set of fields including medicine, environmental science and chemical synthesis. For example, in zymology, quantitative oxygen sensing is required to determine and understand the enzyme kinetics of particular fermentation processes [1].

Typically oxygen sensing, in the liquid phase, can be achieved through the use of modified Clark electrodes [2]. However, there are many limitations associated with the Clark electrode including long – term sensitivity, as the active electrolyte within the probe is consumed over time, and potential electrical interference issues [2]. Furthermore, interrogation is unsuitable in flammable environments.

Optical fiber and integrated optics chemical sensors overcome these limitations of electrical signal based sensing methods because optical fibers are relatively inert to environmental noise and no spark risk.

B. Bragg grating chemical sensors

It has been previously shown that an optical waveguide containing a Bragg grating is an effective method of detecting minute changes in refractive index [3]. Removal of the part of the waveguide's clad allows the evanescent field of the optical mode to interact with an analyte.

The device works by detecting the associated effective index change when the analyte medium is altered. The change in effective index across the Bragg grating causes a shift in the Bragg wavelength of the grating. The relationship between effective index, n_{eff}, and Bragg wavelength, λ_B is shown in Equation (1),

$$\lambda_B = 2\,n_{eff}\,\Lambda \qquad (1)$$

where Λ is the period of the Bragg grating. For a silica waveguide with a Bragg grating in the telecoms region, Λ is ~ 530 nm.

II. FABRICATION OF PLANAR BRAGG GRATING SENSORS

The waveguides are fabricated in flame hydrolysis deposited layers of silica. Doping of the core layer allows a channel waveguide to be defined using a process known as Direct Grating Writing is a UV laser technique that allows simultaneous definition of channel waveguide and Bragg grating. This system differs from conventional direct writing by using an interferometer to generate a ~6 micron spot with a periodic interference pattern. Precise temporal modulation of the UV beam results in the generation of a periodic refractive index with the waveguide. Reference [4] provides a thorough review on the fabrication and development of planar Bragg grating sensors.

An aqueous solution of hydrofluoric acid is used to etch a "window" into the overclad (see Figure 1) and thus exposing the Bragg grating to the surrounding environment [3]. In addition, a ~ 50 nm thick layer of tantalum pentoxide is sputtered on to the exposed Bragg grating. The thin layer of higher refractive index tantalum pentoxide (relative to silica) "pulls" the evanescent wave of the mode into the analyte medium and thus increases the sensitivity of the device [3].

The Planar Bragg grating devices are "pigtailed" to polarizing maintaining fiber and typically interrogated using a broad band optical source and an optical spectrum analyzer.
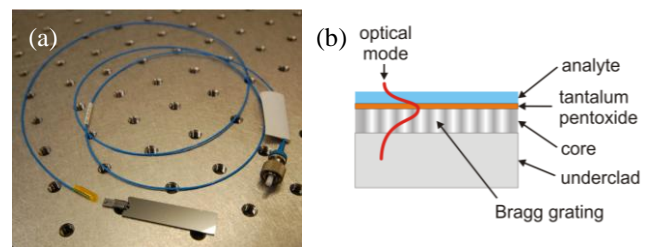

Figure 1. (a) Photograph of a pigtailed etched Bragg grating device (b) Schematic of a chemical sensing integrated planar Bragg grating waveguide

III. INTEGRATED BRAGG GRATING OXYGEN SENSOR

Cao *et al.* has previously demonstrated an optical fiber based oxygen sensor incorporating methylene blue (MB) in a silica sol – gel [5]. It was shown that the absorption spectra of an evanescent field changed relative to the concentration of oxygen passed over the fiber. Therefore it was proposed that a

planar integrated Bragg grating could be used to interrogate MB in a similar way. Replacement of the overclad layer above the Bragg grating with a silica sol–gel, containing immobilized MB, could be used as oxygen sensor in gaseous and liquid phases. This device differs from Cao's system as it detects the change in refractive index of MB.

A. Methylene blue

MB is an organic dye that forms intense blue colored solutions. Amongst other uses, MB is the main reagent of the notable "blue bottle" experiment in chemistry; the oscillating reaction between methylene blue and its reduced form, *leuco*-methylene blue (LMB) [6]. The oxidation of LMB to MB, due to ambient oxygen present, exhibits an explicit color change from colorless to blue, as shown in Figure 2.

B. Silica sol- gels

The photochemistry of MB in a silica sol-gel matrix has been investigated [7]. Silica sol-gels are inorganic materials that consist of a porous silica matrix [8]. Thin glass films of silica sol-gel can be formed through conversion of the initial colloidal suspension of silica, known as a sol, into thin layers of a viscous silica gel by spin or dip-coating. Additional heat treatment promotes further densification into solid materials [8]. Examples of these silica sol-gel thin glass films have been used in optical applications.

IV. PRELIMINARY RESULTS

It was believed that the significant color change upon oxidation of an aqueous solution of LMB to MB would have an associated change in refractive index, detectable by a Bragg grating sensor. The sensor consisted of four Bragg gratings, three etched sensor gratings and one buried reference grating.

A blue colored aqueous solution of MB (of concentration 3×10^{-4} mol/L) was reduced to colorless LMB in a nitrogen environment. An exposed planar Bragg grating device was immersed in this stirred solution and before exposing to a restricted flow of air. Gradual color change from colorless to blue was observed, suggesting that LMB was oxidized to MB, confirmed by UV-visible spectroscopy. The average Bragg wavelength shift monitored on oxidation is shown in Figure 3.
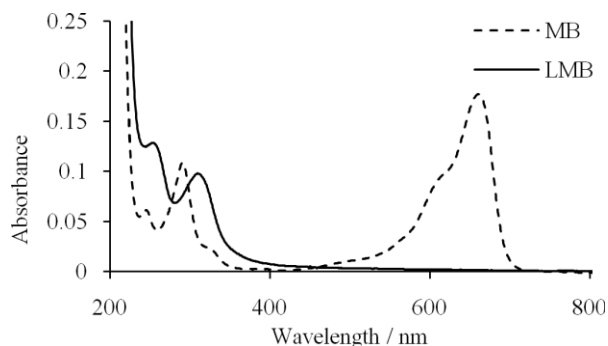
Figure 2.   UV-visble spectra comparing methylene blue and leuco-methylene blue showing the loss of the absorption peak at ~660 nm.
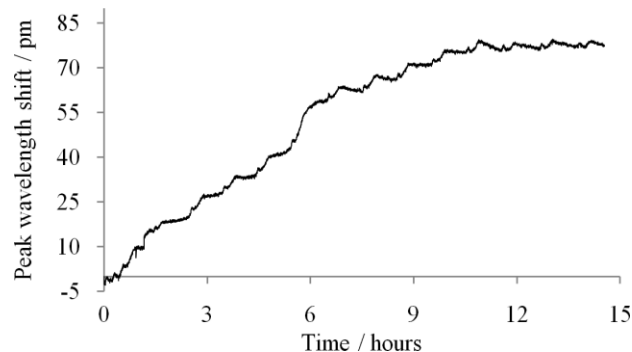
Figure 3.   A shift of ~80 pm in Bragg wavelength during the oxidation of aqueous *leuco*-methylene blue to methylene blue was observed when exposed to a restricted flow of air. This demonstrates the high sensitivity to changes in oxygen concentration of the simple Bragg device. The data is corrected for temperature variation.

A Bragg wavelength shift of ~80 pm, corresponding to an effective index change of ~ 7×10^{-5}, was observed during the oxidation of LMB to MB. The restricted air flow allowed for gradual oxidation over ~12 hours, highlighting the sensitivity to subtle changes in oxygen concentration. Temperature compensation was achieved using a localized buried Bragg grating within the device and a scaling factor of 0.7 was applied to account for the different thermo-optic coefficients for silica and aqueous solvent.

V. CONCLUSIONS:

Here we show preliminary results that a planar Bragg grating can detect changes in the refractive index of methylene blue in solution on exposure to oxygen. We will further develop Bragg grating sensors by immobilizing methylene blue into a sol-gel overlayer that will enhance sensitivity and allow for gas phase sensing. We shall present our latest work towards the production of a robust and reversible oxygen sensor.

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