To: CLEO/Pacific Rim 2001

# Integrated Optical Dual Mach-Zehnder Interferometer Sensor

Ping Hua<sup>\*</sup>, Kenji Kawaguchi<sup>†</sup> & James S. Wilkinson<sup>\*‡</sup>

\*Optoelectronics Research Centre, University of Southampton, Southampton, Hampshire, SO17 1BJ, UK. \*Kyoto Electronics Manufacturing Co., 68 Ninodan-cho, Shinden, Kisshoin, Minami-ku, Kyoto 601, Japan \*Tel: +44 23 8059 2792 Fax: +44 23 8059 3149 Email: jsw@orc.soton.ac.uk

## Abstract

A fibre-pigtailed dual-sensitivity integrated optical Mach-Zehnder Interferometer sensor chip for liquid analytes is described. Thin overlays of  $Ta_2O_5$  and reference structures have been integrated to enhance sensitivity and compensate for drift and attenuation.

## Introduction

Integrated optical transducers for real-time measurement of interactions between biological molecules and for the specific detection of chemical and biochemical species are the subject of growing interest. The increasing complexity of applications in pollution monitoring and medical diagnostics demands integration, so that approaches that allow interrogation of arrays of sensors on the microstructured surfaces, where small volumes of analyte are controlled by integrated microfluidic systems, are expected to find wide application. The integrated optical Mach-Zehnder interferometer (MZI) may be used as an evanescent refractometer and may be chemically modified to render it sensitive to specific chemical species. Highly sensitive MZI immunosensors have already been demonstrated, for example by Heideman et al [1], and our work has concentrated upon achieving sensitive operation over a wide range of indices, enhancing the sensitivity of ion-exchanged devices using high-index overlayers, and investigating the simple and reliable incorporation of these sensors instrumentation. The design adopted into incorporates i) thin tantalum pentoxide films to enhance sensitivity, ii) a high-sensitivity MZI and a low-sensitivity MZI to allowed the combination of high sensitivity with wide index range, iii) 3x3 output couplers from each MZI to ensure sensitive operation over the entire index range and to remove ambiguity in the direction of index change, and iv) reference waveguides with and without analyte windows, to allow reduction of the effects of input power fluctuations and potentially to determine analyte absorption. In this paper we present multiple-output integrated optical sensor devices using fibre input coupling and a cheap, readily available, 1D CCD array detector to simultaneously address all outputs.



SUGA

### **Device Fabrication**

The transducer chip shown in Figure 1 was fabricated by ion-exchange in BK7 glass. The substrate was masked with Al patterned with 2µm wide waveguide openings, and immersed in KNO<sub>3</sub> at 400°C for 2 hours; the ends of the chip were then polished to allow input and output coupling, resulting in an overall length of 44mm. A silica isolation layer of thickness 1µm was sputtered onto the chip, with windows of 8mm 0.8mm length being defined and photolithographically by positive lift off. The device was then annealed in air at 250°C for 1 hour. A tantalum pentoxide film was deposited over the entire surface of the chip by reactive sputtering from a tantalum target. This film was deposited in three stages (each of thickness and sensing measurements were  $15 \pm 1$  nm) conducted before and after each stage, so that results were obtained for the same MZI's with a Ta<sub>2</sub>O<sub>5</sub> overlayer of nominal cumulative thickness 0, 15, 30 & 45nm. The final device was then annealed in air at 250°C for 1 hour, and the measurements repeated.

#### Operation

In each interferometer, the relative phase of the light in the two paths combining at the 3x3 coupler depends upon the refractive index of the analyte in the window in the isolation layer. The use of three-waveguide couplers results in three outputs, with interference functions shifted with respect to each other. In this way at least one output yields a sensitive response to small index changes whatever the baseline index [2]. One MZI has a window ten times shorter than the other, resulting in ten times lower sensitivity, to

remove ambiguities for large index changes due to the periodic nature of the index response. Two reference outputs are provided, one isolated from the analyte for its entire length for removal of the effects of input power fluctuations, the other exposed to the analyte through an identical window to that of the high-sensitivity MZI, yielding a direct measurement of the loss due to the window and analyte.

#### Measurements

Light from a 10mW 633nm He-Ne laser was coupled into a single-mode polarisationmaintaining fibre, which was butt-coupled to the sensor chip. A preliminary inspection confirmed that the waveguides were monomode at this wavelength. A x10 objective lens was used to focus the output signals onto a 1D CCD array. A 40dB neutral density filter was placed between the lens and the detector to prevent saturation of the CCD array, and a sheet polariser was used to select the TE polarisation. A flow-cell was clamped to the sensor surface and test solutions of sucrose in water of refractive indices between 1.3330 and 1.4505 were applied using a flowinjection system. Figure 2 shows the phase change introduced in the high-sensitivity MZI against refractive index relative to water, over the range of sucrose solutions tested, for the 0, 15 & 30nm  $Ta_2O_5$  thicknesses. Addition of the  $Ta_2O_5$  film to the high-sensitivity MZI has significantly increased the sensitivity of the device in an aqueous medium. These sensitivities range from approximately 2000° per unit refractive index (RIU) for the uncoated device to 5500°/RIU and 12000°/RIU for the 15 and 30nm coated devices representing enhancement by a factor of 2.75 and 6, respectively. The low-sensitivity MZI with the 45nm Ta<sub>2</sub>O<sub>5</sub> film showed a phase sensitivity of 17,500°/RIU, corresponding to an enhancement greater than 85 for the high-sensitivity MZI, for which detailed results will be presented. Figure 3 shows the attenuation due to the 8mm analyte window (Ref<sub>1</sub>) against index for each Ta<sub>2</sub>O<sub>5</sub> thickness and after annealing of the final device. This loss includes the transitions into the window and the propagation loss in the Ta<sub>2</sub>O<sub>5</sub> coated region. Clearly the loss becomes significant for the sample overlaid with  $30nm Ta_2O_5$  and increases with increasing analyte index as the modal fields in the Ta<sub>2</sub>O<sub>5</sub> coated region are drawn towards the waveguide surface. This increases the modal mismatch between the coated and uncoated regions and causes more power to propagate in



Figure 2. Phase response of high-sensitivity MZI the relatively lossy  $Ta_2O_5$  film, both serving to increase attenuation. Such attenuation causes reduced interference contrast in the MZI's, acting against the increased phase sensitivity with thicker  $Ta_2O_5$  films to reduce the minimum detectable index change. The window attenuation becomes excessive for 45nm  $Ta_2O_5$ , with attenuation of over 30dB for all analyte indices above that of water. Annealing the device reduces the attenuation significantly, by reducing the optical absorption of the  $Ta_2O_5$ , but it is still excessive for optimum operation of the high-sensitivity MZI. The phase sensitivity of the MZI was not measurably changed after annealing.



#### Conclusions

An integrated dual Mach-Zehnder Interferometer sensor chip with wide analyte index range has been demonstrated. High index overlayers serve to increase the phase sensitivity significantly while increasing attenuation. Reference waveguides allow compensation for input power fluctuations and analyte absorption. Analysis of detection limits and optimisation of  $Ta_2O_5$ thickness will be presented.

1 RG Heideman et al., Sens.Actuators, **61**, 100, (1999) 2 BJ Luff et al., J. Lightwave Technol., **16**, 583, (1998)