OPTOELECTRONICS RESEARCH CENTRE

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Introduction

The principal contribution of Southampton University to the BIOPTICAS project is in the realization of planar optical waveguide probes to determine the optical properties of attached sensing films. Three types of device are being investigated: surface plasmon resonance (SPR), directional coupler and chemiluminescence sensors. Techniques have been established for the deposition of compatible electrodes for electrochemical modulation of sensing reactions as an integral part of devices, and equipment has been set up for the fabrication of waveguides in glass substrates by field-assisted ion-exchange. The modelling and design stages for the devices are now close to completion, and we have begun the fabrication and evaluation of preliminary designs and verification of models. Interaction with partners has resulted in the establishment of standardised sensor chip formats and plans for comparative evaluations of the sensors developed in the project, using standardised sensing reactions, are well in hand.

Sensor Modelling and Design

Rigorous and reliable design tools for multilayer waveguide probes of thin sensing films have been established. The multilayer model is based on transfer matrix theory and utilizes the argument principle method (APM) to determine the number of modes which exist in the complex plane of modal velocity and absorption. The model has been extended to include the coupling efficiency of light into and out of the various modes in the multilayer region, using an approach based on the calculation of the overlap integrals between the modal fields of the uncovered guide and those of the multilayer region. This model represents the first full treatment of such devices and experimental tests are under way in order to establish its validity. The model has been applied to the optimization of surface plasmon resonance (SPR) sensor designs both with and without a low-index buffer layer between the metal film and waveguide surface, as shown in Figure 1. In both cases use of BGG31 glass, as supplied and used by IOT, as substrate material offers the highest sensitivity to changes in adsorbed film thickness or index. The simpler design without a buffer layer is viable for the aqueous environment, but use of a low-index buffer layer, as described below, would result in enhanced performance in terms of the normalized change in power transmitted through the device upon addition of a thin sensing film. The latter case is shown in Figure 2, where the effect on transmission of the device upon adsorbing a film of thickness 7nm and index 1.47 is shown as a function of the buffer and gold thicknesses. Attachment of sensing films to these gold electrodes and their use for electrochemical control of sensing reactions, is being investigated in parallel by Liverpool University.

Because of the greater experimental complexity associated with the fabrication of a buried directional coupler (BDC) structure, it has been decided to proceed initially with the design and fabrication of a sensor based on a lateral directional coupler (LDC), which is shown in Figure 3. The basic principle of operation is exactly the same as for the proposed BDC sensor, and the model developed will apply to both structures. The main problem associated with the lateral structure is the need for a cover layer to mask one guide of the coupler so that its properties do not change with superstrate index, in common with the Mach-Zehnder (MZI) sensors produced by IOT. In both cases the second guide has the sensing film overlayer attached and must be exposed to the analyte. To accomplish this photolithographically requires careful mask design and alignment as the separation between the two guides is only a few microns. A CAD-based mask design software package is being used in conjunction with optical waveguide design software, and a suitable multilayer alignment system has been designed. The MZI devices produced by IOT also use a cover layer to isolate one arm of the interferometer. A silica layer is suitable for devices produced in BGG36 glass (n = 1.6) but use of the lower index glass BGG31 (n = 1.47) will require a cover layer with an index lower than that of silica. For this reason, work is being undertaken on the deposition and lithography of MgF_2 (n =1.39) and other thin films having an index of 1.29, for use with LDC, and potentially MZI, devices which use BGG31 substrates. Use of low-index buffer layers for SPR devices is also desirable, as described above. Refractive indices given are approximate and apply in the red and near infrared wavelength regions. Potentially suitable hard, moisture resistant MgF_2 films have been produced by heating the substrate to 100°C during deposition, and methods for patterning these films are currently being assessed. Test samples of polymeric materials in solution for spin coating and evaporation have been obtained. The possibility of adding an extra protective layer above the low index layer (eg. SiO_2) is being considered. Modelling of the directional coupler devices is more complex than for the SPR devices because ion-exchanged channel structures must be fully analysed, and use is being made of a commercially available beam propagation method (BPM) software package (CAOS) specifically designed for the analysis of graded-index channel waveguide structures. The expected change in output power of a directional coupler device as a function of superstrate index is given in Figure 4. Sensitivity to changes in the index of a thin sensing film is to be analysed, and will be compared with the SPR and MZI sensor designs.

A model based upon a plane wave spectrum representation has been developed to predict the efficiency of launching of chemiluminescence into waveguide modes in channel waveguides, from reactions taking place in the analyte close to the waveguide surface. Results using this model indicate that a high refractive index ratio between waveguide core and analyte is a requirement for efficient light collection. More detailed calculations are being carried out to determine the collection efficiency as a function of distance from the waveguide surface so that light collection may be optimized for a given sensing reaction. This will allow design of optimised waveguide structures to complement the spatial distribution of light emission above ITO electrodes being determined experimentally and theoretically at Liverpool University.

Gold & ITO thin-film electrode evaluation

Extremely robust gold layers have been produced by chemical treatment of the glass surface prior to gold evaporation using (3-mercaptopropyl)trimethoxysilane. The films easily pass the simple adhesive tape test and resist removal even after several hours in an ultrasonic bath. Preliminary SPR measurements using a coated prism indicate that the silanization treatment does not affect the resonance curve. SPR waveguide devices are therefore presently being produced using this process.

ITO films with low sheet resistance are readily produced using standard techniques; however, the use of ITO films on waveguides for modulated chemiluminescence collection severely limits the allowable thickness and absorbtance of the films. The films being developed in this project must have thicknesses of 10-30nm, making low sheet resistance a challenging goal. Such ITO films are now being produced that exhibit sheet resistances of 120 - 600 Ω/□ with good transparency (> 90% transmission over the range 0.6 - 2 μm) that are suitable for use as electrodes in modulated chemiluminescence experiments. Previous films have been annealed following deposition at around 200°C in a cracked ammonia atmosphere, but were found to be unsuitable; better films are produced by annealing at higher temperatures (around 300°C) in air. Further optimization of parameters during deposition is expected to lower the required annealing temperature to make the process more suitable for application to glass containing ion-exchanged waveguides. These films will be used for parallel experiments in electrode-loaded waveguide quality, and electrochemiluminescence at Liverpool University.

Field-assisted ion-exchange

Apparatus for controllable, highly repeatable, field assisted ion-exchange in glass substrates has been established. The application of this equipment is principally in the realisation of the buried directional coupler devices by sequential field-assisted ion-exchange steps, and in the burying of waveguides for the control of interaction strength and improvement in sensitivity in SPR devices. The apparatus was designed with the intention of maximum flexibility and reproducibility for the waveguide fabrication which is very important for the fabrication of directional couplers, for example. The process parameters can be controlled directly, or monitored and controlled using relations between the process parameters, for example for ion-current monitoring and control using the high voltage actuator. This offers the possibility of time-dependent current control during the ion-exchange so that process flexibility, especially for the fabrication of directional couplers, can be increased. The substrate and the anode melt are held by a vacuum chuck which is mounted to a motor driven support and can be driven into the furnace with the cathode melt inside. This allows a controlled heating and cooling of the substrate using the temperature profile of the furnace. The process temperature of the anode melt, cathode melt, substrate, and the furnace are monitored using thermocouples. The process temperature which may be achieved is up to 600°C, for samples of diameter up to 40 mm. The voltage which may be applied between the electrodes is limited to 270V over which range a current of at least 70 mA may be delivered. There is computer control of the high voltage unit, and of the motor drive which places the substrate in the correct region of the furnace for the initiation of the process and the control of temperature. The PC also performs continuous monitoring of furnace, melts, and substrate temperatures, and ion-exchange voltage and current.

Conclusion

Work is proceeding on the three types of optoelectrochemical sensor proposed in this programme, for application in the detection of pesticides in ground water. Models of each type of sensor have been developed and techniques essential for the realization of the devices have been established or are under investigation. Fabrication of initial sensor designs has begun, and experimental results generated by our partners in this project are being used to establish and refine practical sensor designs. Plans have been made for comparative evaluations of sensors using standard sensing reactions established by our partners.

Selected bibliography

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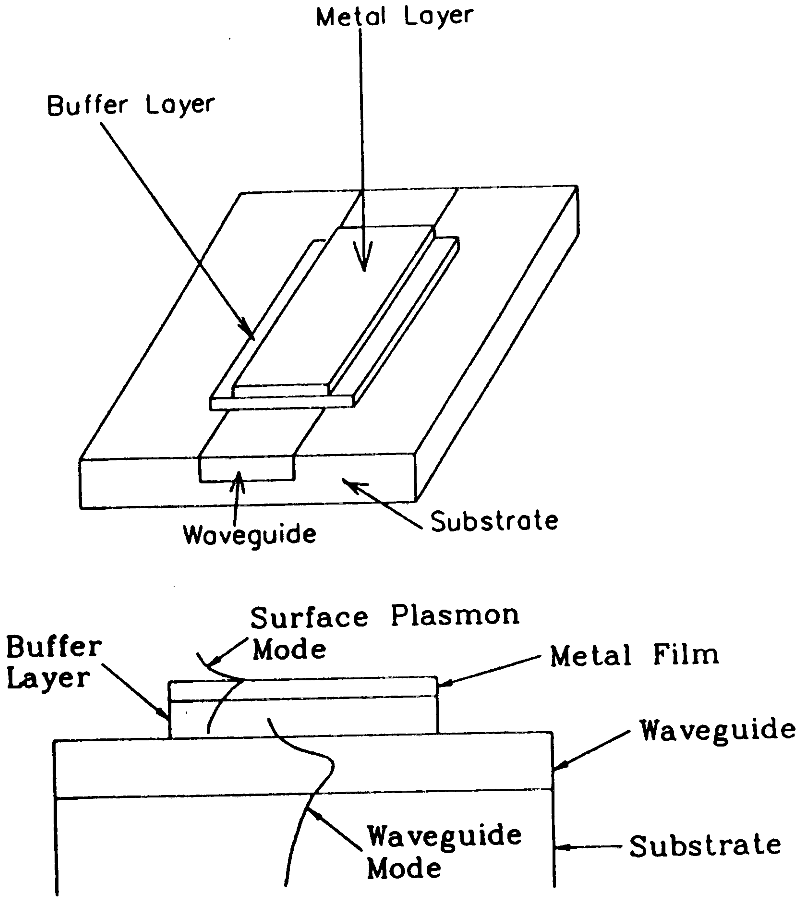
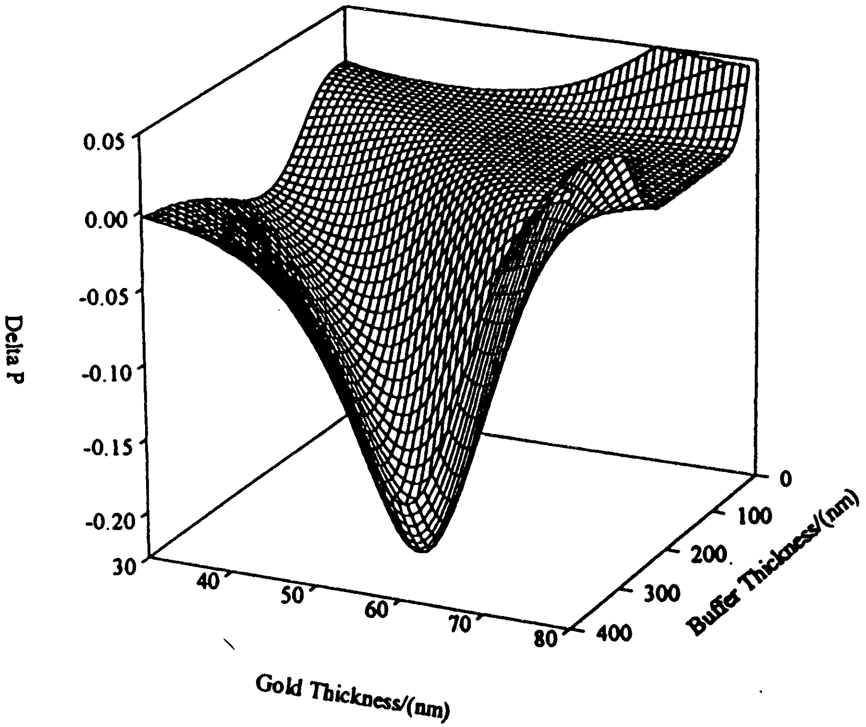


FIG 1: **Waveguide-coupled surface plasmon resonance sensor.**



System Parameters :-
Superstrate Index=1.33
Film Index=1.47
Film Thickness=7nm
Film Length=1.5mm
Buffer Index=1.29
Guide Index=1.47
Guide Depth=2μm
Substrate Index=1.46
λ=639nm

FIG 2: Surface plot showing the change in power transmitted through an SPR sensor on adsorbing a thin film. ΔP is normalised to input power.

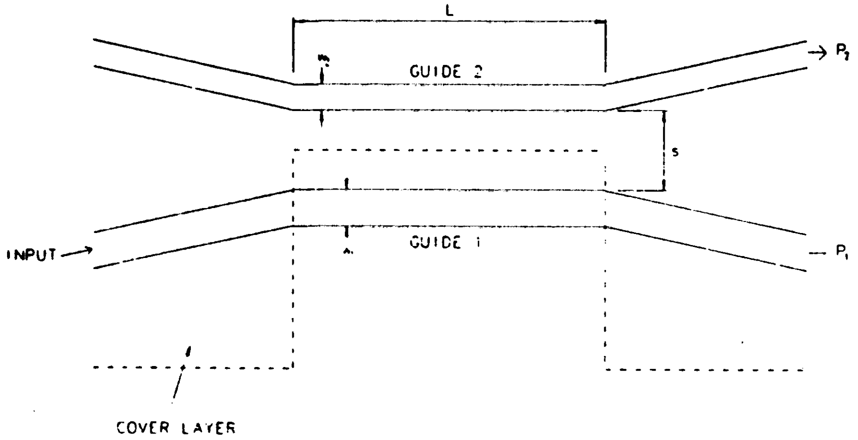


FIG 3: Lateral directional coupler sensor.

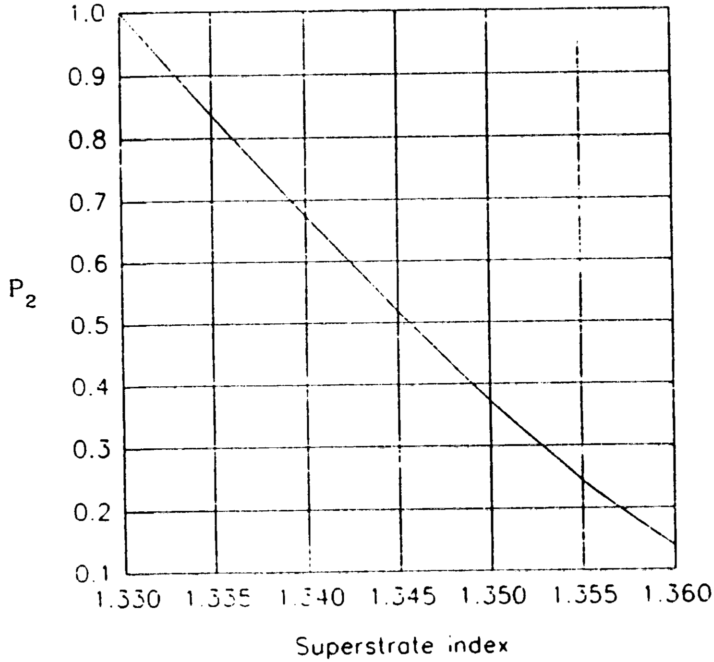


FIG 4: Normalised power output from guide 2 of a lateral coupler as a function of superstrate index. $w_1 = 3\ \mu m$, $w_2 = 2.75\ \mu m$, $s = 10\ \mu m$, BGG31 substrate, MgF_2 cover layer.