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# An optical fibre based evanescent wave sensor to monitor the deposition rate of thin films

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**Abstract**

A novel fibre optic sensor for the in situ measurement of the rate of deposition of thin films has been developed. Evanescent wave in the uncladded portion of a multimode fibre is utilised for this sensor development. In the present paper we demonstrate how this sensor is useful for the monitoring of the deposition rate of polypyrrole thin films, deposited by an AC plasma polymerisation method. This technique is simple, accurate and highly sensitive compared with existing techniques. © 1998 Elsevier Science S.A. All rights reserved

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## 1. Introduction

Fibre optic sensors (FOSs) play a major role in the field of photonics as applied to research and industry. Many of their attractive features have caused them to earn a prominent place in the forefront of photonics technology. Recent works in this emerging area prove that the FOSs have a remarkable ability to measure physical and chemical variables with unprecedented sensitivity and accuracy [1–4]. Also, there exists enormous potential for FOSs to be developed as an important technique for on-line measurements. Quite a few FOSs have been developed in recent times for the monitoring of a variety of physical parameters [5]. The present paper discusses the design and implementation of a simple FOS for monitoring the deposition rate of thin films.

Different techniques such as resistance monitors, capacitance monitors, quartz crystal monitors, radiation absorption methods, photometric methods etc., are used at present for the deposition rate monitoring [6]. Each method is well known for its merits and demerits. The film resistance method is applicable only to metallic and low resistivity semiconductor films. For structurally discontinuous films ($<$10 nm), this method cannot be used. The capacitance monitor method is not very sensitive and it may be subject to spurious effects due to stray electrical charges in the vapour and in the vacuum chamber. The maximum sensitivity of a quartz crystal monitor is limited by variations in the crystal frequency due to temperature, oscillator drive level, and changes in the oscillator circuit. Use of suitable radiation shields and also water cooling of the crystal holder are needed in this case. In the radiation absorption method, the absorption in discontinuous films is strongly influenced by the granular nature of films, but it may still be used for relative measurement of the average film thickness. The photometric method uses the oscillatory nature of the interference effect of a reflected or transmitted beam from the film–substrate combination during deposition. This method is mainly used for monitoring and controlling the deposition of multilayer dielectric films. Optical methods have the advantage that they are generally non-contact in nature and hence in situ measurements can be performed very conveniently without breaking the vacuum. Optical fibre based techniques have the added advantage that the possibility of remote measurement is quite inherent.

Although, this paper deals with the measurement of the deposition rate of thin films of an organic material (polypyrrole) which is produced by AC plasma polymerisation, the technique described here can be applied in general to any deposition method.

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## 2. Theory

It is well known that the guided light in a fibre waveguide penetrates into the cladding to a distance of a few wavelengths as the evanescent tail of the waveguide mode. The power transmitted by an optical fibre, the cladding of which has been replaced locally by an absorbing medium may be written as [7]

$$P(z) = P(\mathrm{o})\exp(-\gamma z) \tag{1}$$

where $z$ is the length of the uncladded region of the fibre, $P(\mathrm{o})$ is the power transmitted in the absence of an absorbing species, and $\gamma$ is the evanescent absorption coefficient. The coefficient may be related to bulk absorption coefficient $\alpha$ when all bound modes are launched. In this case the fraction of power ($r$) outside the core is given by

$$r = \frac{4\sqrt{2}}{3V} \tag{2}$$

where $V$ is the normalised frequency parameter of the fibre. For a fibre of core radius $a$ and numerical aperture $NA$, $V = 2\pi a/\lambda\ NA$, where $\lambda$ is the wavelength of the propagating light. Hence Eq. (1) becomes

$$P(z) = P(\mathrm{o})\exp(-r\alpha z) \tag{3}$$

so that the evanescent absorbance, $\log P(\mathrm{o})/P(z)$, of an uncladded fibre of length $L$ surrounded by a medium of bulk absorption coefficient $\alpha$ is given by

$$A = \frac{\gamma L}{2.303} = \frac{r\alpha L}{2.303} \tag{4}$$

where $A$ is proportional to $\lambda\alpha L$/a $NA$. The above equation predicts that the evanescent absorbance depends linearly on the exposed fibre length $L$, the bulk absorption coefficient $\alpha$ and inversely on the numerical aperture ($NA$).

In the absence of evanescent wave absorption, the guidance of light through the uncladded region gets modulated by the changes in the refractive index of the coupling medium. The input optical power coupled through an uncladded region of the fibre to the output port would vary with the dielectric constant ($\in_{\mathrm{m}} = n_{\mathrm{m}}^2$) of the medium surrounding the uncladded region. For a Lambertian source, the output power is given by [8]

$$P = P(\mathrm{o})\frac{n_1^2 - n_{\mathrm{m}}^2}{n_1^2 - n_2^2} \tag{5}$$

where $P(\mathrm{o})$ is the total guided power in the fibre and $n_1$, $n_2$ and $n_{\mathrm{m}}$ are the refractive indices of the core, cladding and the medium surrounding the uncladded region, respectively.

## 3. Experimental details

Polypyrrole is prepared directly from its monomer vapour by a low frequency AC (50 Hz) plasma polymerisation process. A schematic experimental set-up is given in Fig. 1. The sensing fibre with its cladding removed from a region of certain length is passed through the inter-electrode region. A glass substrate on which the thin film has to be deposited is also kept near the fibre. A diode laser (4.25 mW, at 670 nm) is focused onto one end of the fibre and the output power from the other end is measured using a commercial fibre optic powermeter (Meggar OTP 510). First, the system is pumped down to about $10^{-3}$ Torr. Then the voltage is applied between the electrodes to pro-

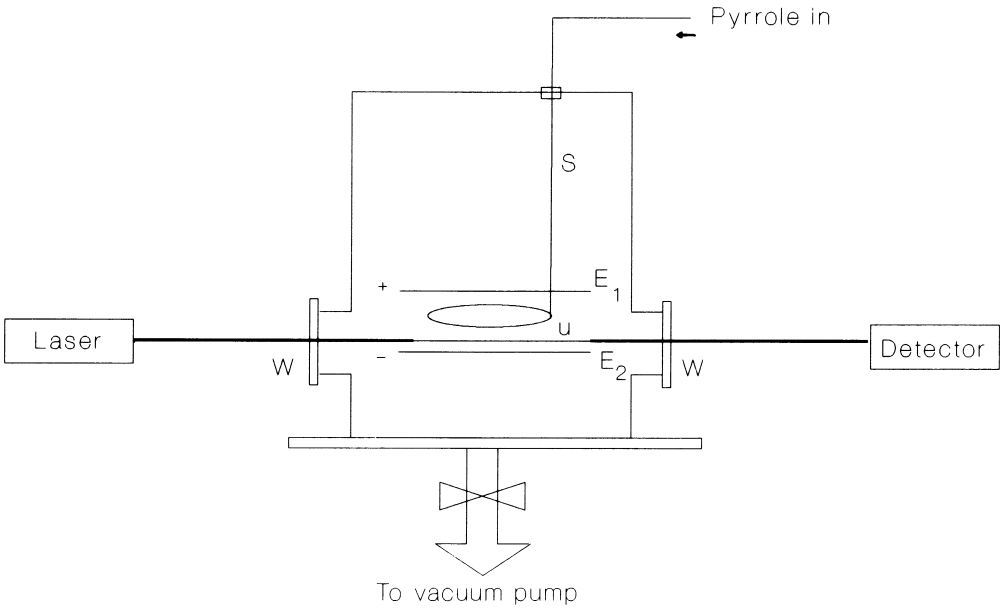


Fig. 1. Schematic of the experimental set-up. W, window; E, electrode; s, sprayer; u, uncladded region of the fibre.

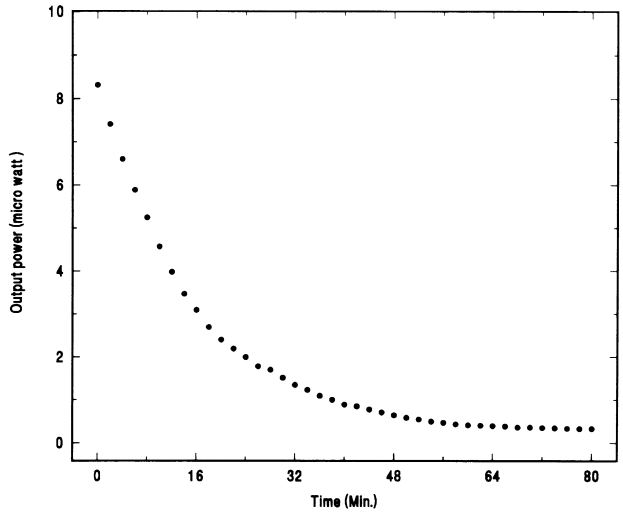


Fig. 2. Typical plot of output power variation with time.

duce the plasma and the monomer is sprayed into the inter-electrode region by means of a sprayer by carefully controlling the flow rate using a needle valve. The pressure ($P$), discharge voltage ($V$), the flow rate ($R$) and the distance between the electrodes ($d$) are adjusted such that a solid coating of polypyrrole is obtained on the glass plate [9] as well as on the uncladded fibre. The evanescent wave coupling of this medium surrounding the uncladded region brings about a change in the output intensity of the fibre as deposition proceeds.

## 4. Results and discussions

The output power from the optical fibre is measured at regular intervals of time during the deposition of film on the uncladded region. Fig. 2 is a typical output power versus time plot for the sensor with an uncladded length of 0.06 m for a particular combination of pressure, voltage, flow rate and inter-electrode distance ($P = 10^{-3}$ Torr, $V = 550$ V, $R$ (position of needle valve) = 70 units, $d = 0.035$ m). As was expected, the graph showed two distinct regions, viz. an almost linearly decreasing region and a saturation region.. From the linear portion of the $P$ versus $t$ graph, $dP/dt$ is calculated. The system is calibrated by measuring the thickness of a few films prepared for different time durations within the linear range. The slope of the thickness ($x$) versus time ($t$) graph yields $dx/dt$. In the linear region, the rate of change of transmitted power is proportional to the rate of deposition.

$$i.e.\ \frac{dP}{dt} \propto \frac{dx}{dt}\ or\ \frac{dx}{dt} = K\frac{dP}{dt} \tag{6}$$

The value of $K$ is evaluated for a set of experimental conditions and is found to be 0.12 nm/$\mu$W. This enables us to calibrate the sensor. Knowing the value of $K$ and by monitoring the output power variation with time, the deposition rate $dx/dt$ and hence the thickness $x$ at any instant $t$ during the deposition can be evaluated.

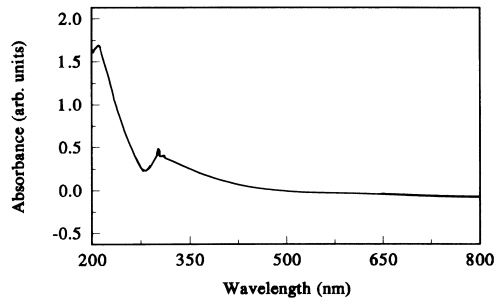


Fig. 3. Absorption spectrum of polypyrrole.

The decrease in the output power with thin film deposition can be attributed either to the absorption of the laser radiation by the deposited polypyrrole or due to refractive index variation. The absorption spectrum of polypyrrole obtained using a Hitachi-340 model UV-VIS-NIR spectrophotometer within the spectral range 200–800 nm (Fig. 3) shows that the material does not have much absorption at the laser wavelength (670 nm) used. So the decrease in output is mainly due to the refractive index variation. That is, as the material gets coated on the fibre, the air cladding gets replaced by the polypyrrole cladding, which has a higher refractive index than air. Due to the decrease in the index difference between the core and the deposited polypyrrole, which acts as cladding, the acceptance angle for the portion of the fibre and hence the numerical aperture decreases and the coupled power correspondingly gets reduced while deposition progresses. When a polypyrrole cladding of a particular thickness is formed, further increase in thickness will not have any effect on the output power, and so the output power remains constant leading to saturation.

Within the limits of accuracy of thickness measurement, we obtained an output variation of 1 $\mu$W corresponding to 0.1 nm deposition. Keeping the pressure, voltage and sensor length a constant, an increase in the flow rate is found to decrease the output power faster showing a deposition rate increase. This is a confirmation of the observed fact that the deposition rate increases with increasing flow rate, passes through a maximum and then decreases slightly [9].

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