Development of a Fiber Optic Sensor for Online Monitoring of Thin Coatings

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Abstract: ***The thickness measurement of gas, liquid and solid layers is not only important for the basic research on nanoscience but equally valuable in contemporary applied biomedical research. Here, we have developed an optical spectroscopy based technique for the online monitoring of thin films (coatings). A low cost light emitting diode (LED) source combined with a fiber optic bundle and grating based spectrograph have been used to generate white light interferogram. We have monitored online change of refractive index of an air film (~4 µm thickness) with temperature following the change in the intensity profile of the interferogram. A thin film of water between two cover slips (thin glass plates) has also been monitored. We have proposed a schematic for further lowering the cost of the developed instrument for the online monitoring of the coating thickness (semitransparent liquid/gas/solid films) during manufacturing/processing. A brief theoretical analysis on the detection limit of the developed technique has also been discussed in the paper.***

Keywords: Nanoscience, thin film, fibre optic, thickness measurement

1. Introduction

With the advancement of science and technology, the interest in studying thin film surface coatings for wear protection and friction reduction has progressed in scientific community as well as in industry [1]. The ability to construct thin films on a variety of surfaces has multiple biomedical applications. A thin film is basically a layer of material ranging from fractions of a nanometer to micrometers in thickness, which can be formed by deposition using physical or chemical methods [2]. In order to meet different requirements, which include thickness, targeting optical or magnetic properties, biocompatibility; the architecture of the resulting film can be designed with nanometer precision (in cross-section) [3].Thus the need to develop a technique for the online monitoring of the formation of thin film surface coating has emerged.

Generally thin film technologies make use of the fact that the properties of the film can be particularly controlled by the thickness parameter. Previous researches have brought to light the utility of near infrared spectroscopy in the analysis of thin film coating indicating the potential of the method for measuring film coating thickness [4]. The other measurement procedures for the measurement of the thin film thickness are stylus profilometry, interferometry, ellipsometry, spectrophotometry and X-ray microanalysis. Reliable results can be obtained from the above mentioned methods only after a very accurate set of operations, which include instrument setup, critical analysis of the results and suitable calibration [5].

Here, we have developed a cost effective optical fiber based spectroscopic method for the online monitoring of thin film coatings made of semi-transparent gas, liquid and solid materials. A white light emitting diode (LED) source is combined in a home-made optical fiber bundle, where central fiber acts as excitation fiber to transfer light from source LED to the thin film and six peripheral collection fibers carry reflected light from two surfaces of a thin film to a grating based spectrograph. The white light interferogram in the spectrograph reveals the thickness of the film or coating following some theoretical formulation well-described in the literature [6]. In order to test the ability of the developed system for the online monitoring of thickness during manufacturing, we have followed optical thickness of an air film between two cover slips at different temperatures. The scope and the limitation of the developed system have also been discussed.

2. Materials and Method

Spectroscopy based absorbance setup for collecting the spectral response of the thin film in between two cover slips is represented in Figure 1. A spectrograph (from Ocean Optics, Florida, USA, Model No.STS-VIS-L-10-400-SMA) with wavelength resolution of 0.47 nm and a white LED source (from Ocean Optics, Florida, USA, Model No.LSS-LED-3650) with wavelength range 400 to700 nm were used in our study. Lab-grade optical fiber (Fiber Type - UV-VIS) from Thorlabs, USA was used for the transmission and collection of light to and from the sample thin film. As shown in Figure 1 schematically, the light from the source is carried by the single fiber in the middle called excitation fiber and is incident on the cover slip while the six surrounding fibers called detection fibers, which collect the diffused light and send the retro-reflected light back to the spectrograph. The collected spectral response generated in the spectrograph is then transferred to a computer through a USB connection where it is processed and displayed using the software (SpectraSuit) that came with the spectrograph [7].

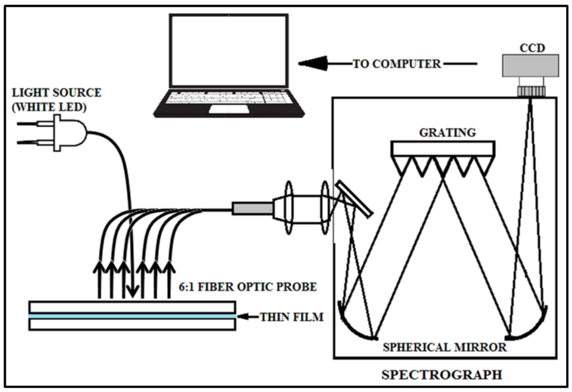

Figure1: Schematic representation of the proposed device. The light from the LED source is transmitted through an excitation fiber and incident on the thin film. The diffused light from the thin film is collected by six detection fibers and transmitted to the spectrograph. The spectral response is processed and generated in the computer.

It is to be noted that the excitation fiber produces a cone of light, which falls on the cover slip surface. The reflected light can be considered to come from a virtual image similar to the thin film formed at the same distance (d) from the cover slips. This phenomenon is illustrated in the Figure 2(a). The reflected light can easily be collected by the detection fibers only when the radius of the excitation fiber is equal to the radius of one of the detection fibers.

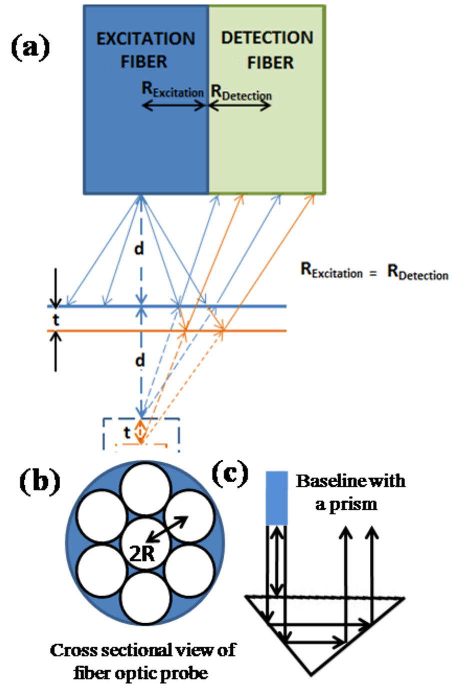

Figure2: (a) The reflected light is assumed to be coming from a virtual source at distance (d) from the thin film phenomenon is possible if radius of detection and excitation probe are equal. (b) The 6:1 fiber optic probe. (c) The principle behind using a prism for setting up of baseline. The arrangement of the fibers in the optical probe is depicted in the Figure 2(b).

In order to set the baseline for the absorption measurement, we have used a right angled prism where only one surface reflected light can be collected (Fresnel reflection) for the better visibility of white light interferogram in the absorption spectrum. It has to be noted that the white light interferogram is the result of the reflected light from a thin film of ~20 micron thickness, because of the limited coherence length of the emitted light from the LED [8]. The thickness of the thin film is measured from the interference fringes generated in the spectrograph. In order to calculate the thickness the equation being used is,

$$t = \frac{M\lambda_1\lambda_2}{2\left[n(\lambda_1)\lambda_2 - n(\lambda_2)\lambda_1\right]} \qquad (1)$$

where, t is the thickness of the film, M is the number of oscillations between two extrema, λ_1 and λ_2 are two wavelengths at two extreme points, $n(\lambda_1)$ and $n(\lambda_2)$ are the refractive indices at the corresponding wavelength. Thus from equation (1) it can be concluded that change in the thickness is not only dictated by the number of interference fringes but also by the refractive index of the material [6].

A thin film of air was taken as the sample in between two cover slips and measurement of the absorbance was carried out at three different temperatures (approximately at 20ºC, 40ºC and 60ºC). Three different interferograms were obtained. The film thickness was calculated using the equation (1) from the collected interferograms. In order to observe the change in the refractive index of the material of the thin film, pure water was taken as the sample in between two cover slips. During the experimental process the thickness of the thin film and the temperature (40ºC) were kept constant.

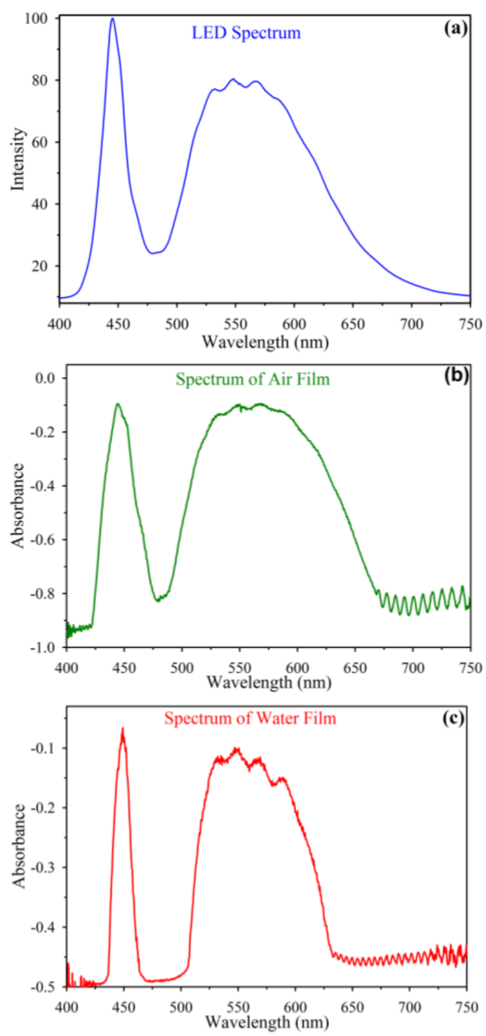

Figure3: (a) Typical spectrum of a white light LED source having peaks at 450 nm and 550 nm. (b) Interferogram of a thin film of air in between two cover slips. (c) Interferogram of a thin film of water in between two cover slips.

3. Result and Discussion

The typical spectrum of a white LED source with peaks at 450 nm and 550 nm is shown in Figure 3(a). Similar spectrum is obtained when a transparent medium is introduced in between the source and the sample. This can be observed when the same LED source is used for obtaining the spectrum of thin film of air and water in between two cover slips. The absorbance spectrum in the region of wavelength ranging from 650 nm to 750 nm displays a number of fringes as shown in Figure 3(b) and 3(c). Different types of information can be derived from this region. The change in thickness and the refractive indices of the thin films according to the variation in the number of the fringes can also be compared.

During the process of deposition of the coating in the form of thin film, the change in thickness can be monitored online from the interferogram. With the variation in temperature the effect on the thin air film is evident from Figure 4(a). Three different fringe patterns were obtained at three different temperatures (at 20ºC, 40ºC and 60ºC). Calculations based on equation (1) showed the different thickness at respective temperature. The graph in Figure 4(b) shows how the change in thickness is almost linear

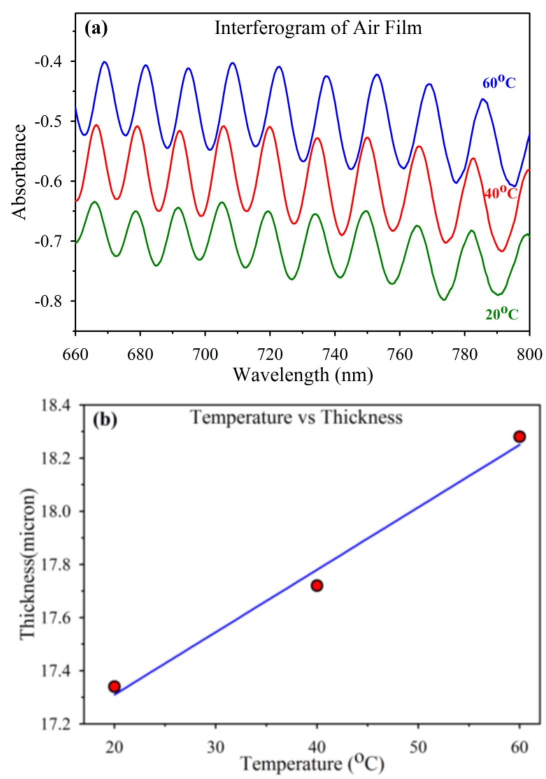

Figure4: (a) The various fringe patterns of the thin air film obtained at different temperatures. (b) The variation of thickness of the thin film with changing temperature.

with the change in the temperature. Thus it can be concluded that with the increase in temperature there is an increase in the thickness of the thin film of air. According to the Charles Law the volume of an ideal gas is proportional to the absolute temperature if the pressure is constant. Here, considering the atmospheric pressure as the constant pressure, the volume of the air film increases as temperature increases because the molecules of the gas have more kinetic energy (that means they are moving faster) and strike the surface of the cover slips. Since the volume is free to increase (non-confined space) the increase in thickness is a result of the increased temperature. Remarkable difference in the fringe patterns of the interferogram was obtained with the change in the refractive index of the material forming

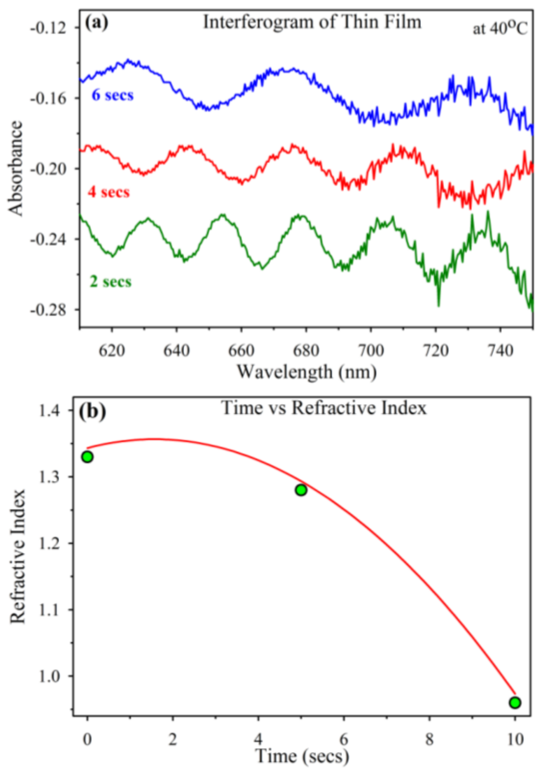

Figure5: (a) The change in the number of fringes in the spectrum of the thin film with respect to time at 40°C. (b) The variation of refractive index (the conversion of water film to air film in between the cover slips) of the thin film with time.

the thin film. It can be concluded from Figure 5(a) that as the refractive index is decreasing there is a decrease in the number of fringes if compared with respect to the same range of wavelength. At a particular temperature the variation in the refractive index of a thin film of water with respect to time is shown in the Figure 5(b). Here we have considered the film thickness of water in between two cover slips to be 20 μm. We have validated the thickness of the water film using UV-VIS absorption spectrometry. We have recorded the absorbance spectra of crystal violet solution of a particular concentration in Shimadzu UV-2600 spectrophotometer. We have repeated the experiment on different sets of glass slides pair and have recorded respective absorbance spectra. The optical path length i.e. the film thickness (l) has estimated with known concentration (c) and extinction coefficient (ε) [9] and the absorbance (A) of the crystal violet using the following equation, and found to be (19.6 ± 1) μm at 40°C.

$$l = \frac{A}{\varepsilon c} \qquad (2)$$

This graph exhibits a nonlinear response which is parabolic in nature.

4. Conclusion

We have developed a spectroscopic technique using a simple fiber optic setup, which is very useful for online thickness and refractive index measurement of the thin films or thin coatings. The developed technique is inexpensive, feasible and accurate. We have demonstrated that our developed technique can also detect very small change in the thickness of the thin films with respect to temperature variation. On the other hand at any particular temperature, we can monitor the difference in refractive index with time. In addition to the above features, the developed technique also offers the scope of future development for more information about the thin films, like changes in the properties during the formation of the thin film coating.

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