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Opto-electronic humidity sensor with incident light in the form of a conical beam

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In the present work, an extensive experimental investigation of TiO_2 film has been carried out. TiO_2 films have been deposited on a prism base by using sol-gel spin process. As the humidity increases in the environment where such films are placed, physisorption and chemisorption of water take place through the pores of films. This leads to a corresponding modulation in the intensity of reflected light. Thus, relative humidity is estimated directly in terms of modulation in the intensity of light observed on a digital power meter. Surface morphology of TiO_2 films has also been studied.

Keywords: Humidity Sensor, Thin Film, Conical beam

IPC Code:G02B

1 Introduction

Monitoring and control of humidity are becoming increasingly important with progress in industrialization. Depending upon the requirements, humidity sensing has been carried out by different kinds of sensors. Optical humidity sensors play a very important role where, besides requiring high sensitivity, the sensor is also required to have remote analysis capability as well as electromagnetic disturbance free monitoring. There is, therefore, an ever-increasing need to develop cheap, robust and highly sensitive opto-electronic humidity sensors, which can be adopted to meet these requirements.

Several opto-electronic refractometers have been developed in our research laboratory[1-3]. They have no moving parts and are sensitive over a wide range of refractive index measurement range. The refractometer described in Ref. 2 is based on the detection of modulation in the intensity of a conical light beam. Angle of incidence at the glass-ambient interface in this configuration varies continuously over a range depending on the angle of divergence of the conical beam. Reflected light is converged at a point for measuring its intensity with the help of detector. This configuration has been modified in Ref.3 by introducing a novel feature in which a thin circular plate having pinholes along its diameter placed in the emergent light beam enables separate detection of light beams reflected at different angles at the glass-ambient interface. Manifold enhancement in sensitivity has been reported for a refractometer configured in this fashion. In the present paper, we report a humidity sensor in which this newly designed refractometer[3] is coupled to a humidity sensitive[4-34] thin film of titanium dioxide deposited by sol-gel spin process.

2 Principle of Operation

The ratio of reflected (I_r) to incident (I_i) light intensities from the interface between two dielectrics for an unpolarised light beam incident at angle θ_i is obtained[2] from the Fresnel equation[22]. Variation of this ratio (I_r/I_i) with refractive index (μ_a) of the second dielectric (ambient medium in our case) for a given μ_g shown in Fig. 1. μ_g is the refractive index of the first dielectric, which is glass prism in our case, forming the interface. In Fig. 1, up to point a, which corresponds to critical angle, total internal reflection takes place. Beyond this I_r decreases as μ_a increases (a to b to c), going to zero at $\mu_g = \mu_a$ (point c). Thereafter, I_r starts increasing with increase in μ_a.

Modulation in reflected light intensity with change in refractive index μ_a is termed as refractometer sensitivity and represented by[1]:

$$S=[\Delta(I_r/I_i)]/\Delta\mu_a$$

Thus, the refractometer sensitivity is maximum in that region of curve (Fig. 1) where the modulation in reflected intensity is maximum i.e. the region a to b. When a thin film of TiO_2 is deposited on the base of glass prism, the glass-film interface behaves like a

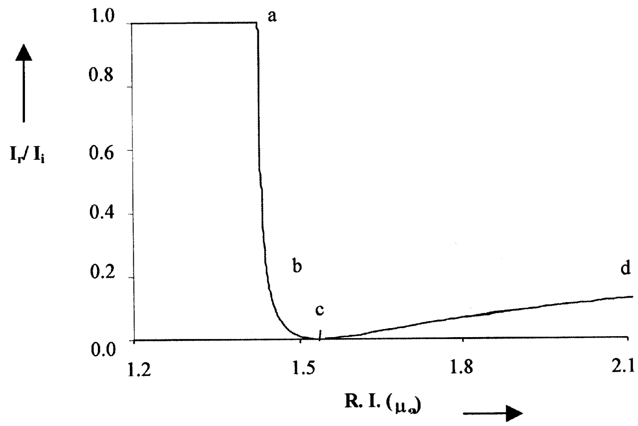

Fig. 1—Characteristic curve showing variations in the ratio I_r/I_i with those in μ_a at an angle of incidence θ_i = 67.6° for μ_a =1.522

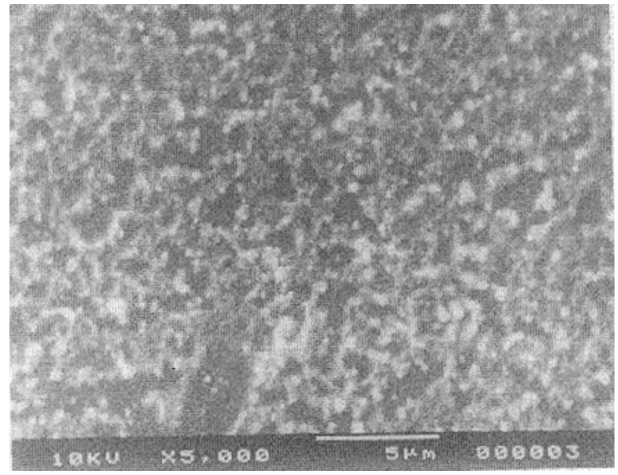

Fig. 2—SEM image of the one layered film of TiO_2

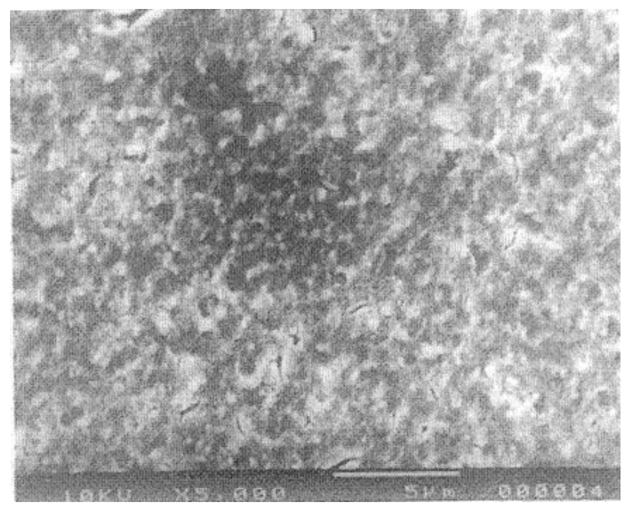

Fig. 3—SEM image of the two layered film of TiO_2

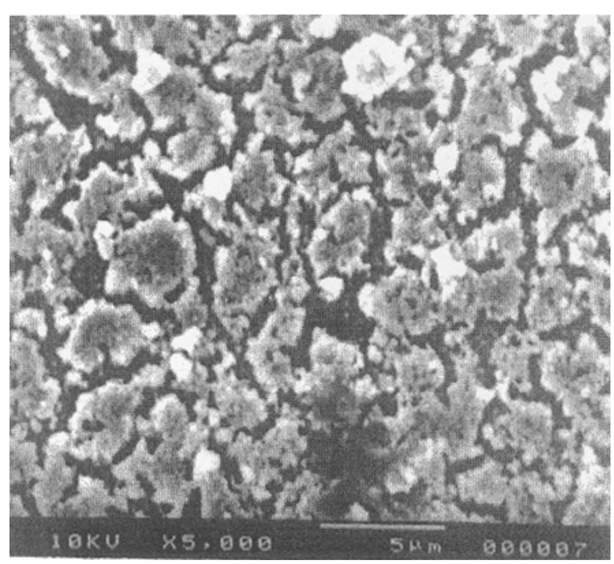

Fig. 4— SEM image of the three layered film of TiO_2

humidity sensor. Configurations with one and two layers of TiO_2 deposited by sol-gel spin process have been studied.

3 Film Depositions

To start with one to four layers of thin films of TiO_2 have been deposited on the base of separate glass prisms (refractive index =1.522, base angles of 67.6° each and base length = 2 cm) by sol-gel spin process. The sol-gel is prepared by mixing isopropyl titanate and isopropyl alcohol[24] in the ratio of 2:25. The spinning speed has been kept at 3000 rev./min and timer has been set at 30 seconds.

Each of the films is dried for half an hour and then annealed at 450°C for four hours so that the TiO_2 film turns rutile[16] and becomes sensitive to humidity. All the films show good adhesion to substrate glass. Surface morphology of one layered, two layered, three layered and four layered film have been shown in Figs 2-5, respectively. A close look at their microstructure shows that the film with two layers of TiO_2 is structurally more porous and uniform than those with one or three and four layers of TiO_2 in which molecules of TiO_2 combine with each other to form clusters with several cracks. We have therefore studied here only the films with one and two layers of TiO_2 deposited on them.

4 Sensor Configuration and Experimental Details

Fig. 6 gives the view of the configuration used. Parallel light beam from a monochromatic unpolarised, 2 mW He-Ne Laser [Model 1303 SRL No. LH 9746 OSAW (India)) of wavelength 630] nm passes through a combination of two microscope-objectives of magnification 45X and 20X producing a

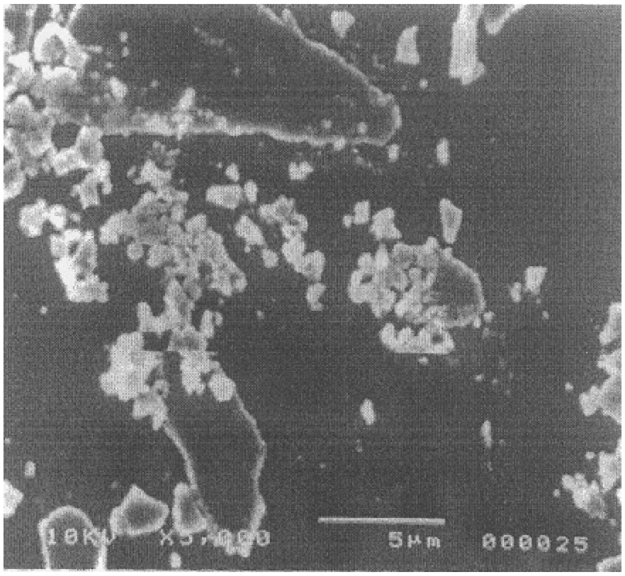

Fig. 5—SEM image of the four layered film of TiO_2

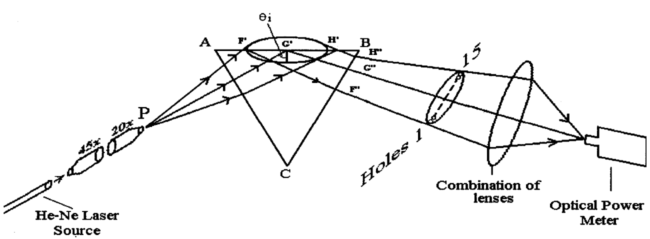

Fig. 6—Experimental set-up

divergent beam of semicone angle 23.8°. In a symmetrical situation, when the central ray PG of this beam cone falls at right angle on the isosceles face AC of the prism, the light spot FH on this surface is circular and that given by F'H' on the μ_g-μ_{film} interface is elliptical in shape[2]. The angle of incidence θ_i on the major axis F'H' of this ellipse increases continuously with the smallest angle lying on the point F' on the largest on H'. The spot formed by the emergent light beam is again circular in shape. The light beam is reflected from the glass-film interface. For a given divergence of the incident conical beam, the diameter of the emergent spot at a particular distance is fixed. A mask of diameter equal to that of the spot is taken in which several holes at equal distances are drilled along the horizontal diameter of the emergent spot. Each hole corresponds to approximately a particular angle of incidence at the glass-film interface. At a time, only a single hole is opened keeping other holes masked. Thus, the reflected rays corresponding to approximately a single angle of incidence are isolated which are collected through a lens combination and focussed on to a power meter (Bench Mark Model FOPM -101).

Saturated solution of potassium sulphate is used as humidifier of the chamber and saturated solution of potassium hydroxide as its dehumidifier. The intensity of reflected light I_r from the glass-film interface is recorded as relative humidity (RH) inside the chamber slowly increases up to 95%. In the process the temperature[24] of the chamber decreases by 4°C. The chamber is then dehumidified firstly up to 10% RH by using the dehumidifier and then up to 5% RH by carrying out the heat cleaning cycle of the sensing element. The least count of calibrating hygrometer used here is 1% RH and that of the optical power meter is 0.1 dBm.

In our experiment, fifteen holes each of diameter 1.0 mm were drilled over a diameter of 2.3 cm of the mask. As the diverging light refracts through the isosceles face of the prism through which it enters, the original divergence of 47.6° gets reduced to 30.8°. Holes 1 to 15 of the mask correspond to the angles of incidence θ_i = 52.2°, 54.4°, 56.6°, 58.8°, 61.0°, 63.2°, 65.4°, 67.6°, 69.8°, 72.0°, 74.2°, 76.4°, 78.6°, 80.8° and 83.0°, respectively on the glass-film interface. For each hole, the output intensity is measured for one layer as well as two layers of the film deposited separately on two prisms. Some representative curves for I_r versus RH% are given here. Fig. 7 corresponds to the case of single layer of the film and Fig. 8 corresponds to the double layer.

5 Results and Discussion

Variations in the reflected light intensity I_r as RH increases from 5% to 95% have been given in Figs 7 and 8, for one layered and two layered films, respectively. The results are found to be reproducible on repeating the experiments. Experimental results which are explained on the basis of Fresnel reflection, agree with the theory, as shown by few representative curves in Figs 9-11. For single layered film as the relative humidity increases, normalized output power decreases at angle of incidence θ_i = 56.6°(Fig. 9), whereas for angle of incidence θ_i = 67.6°(Fig. 10) the normalized output power increases. Theoretical and experimental values are very close to each other. Fig. 11 shows the normalized output power with the variation of relative humidity at an angle of incidence θ_i = 56.6° for two layered films of TiO_2. Theoretical and experimental curves are nearly similar. Of all the configurations the highest sensitivity for entire range

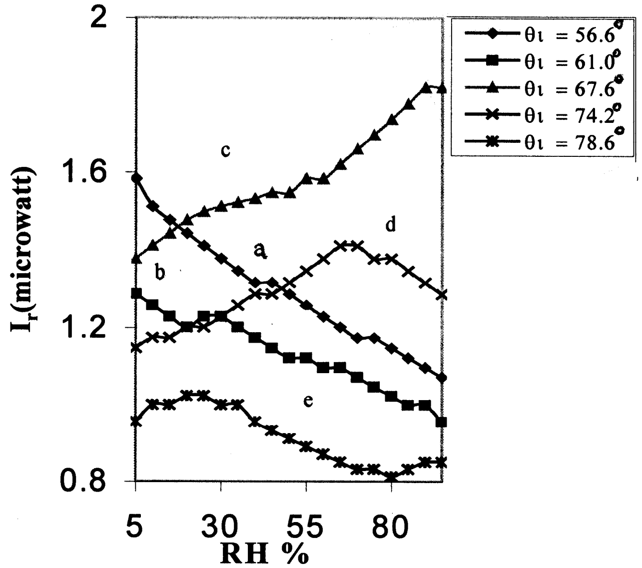

Fig. 7—Variations in the reflected intensity of light I_r against RH% with one layer of TiO_2 deposited

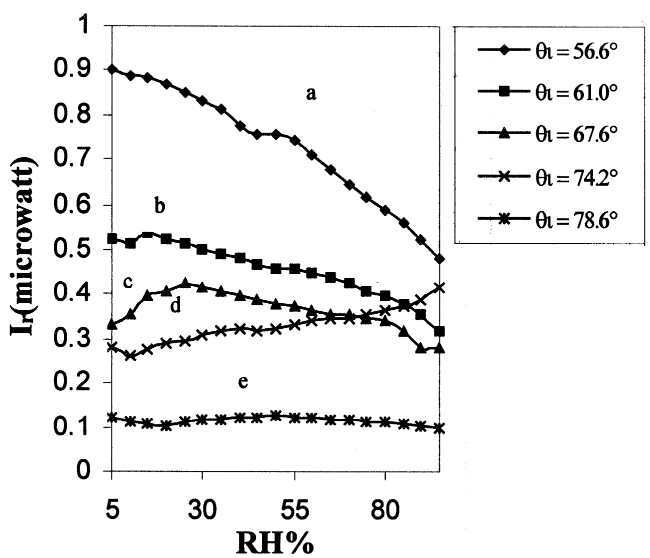

Fig. 8—Variations in the reflected intensity of light I_r against RH% with two layers of TiO_2 deposited on the base of prism

of humidity occurs for one layered film at θ_i = 56.6° corresponding to hole no.3.

Initially, the TiO_2 film is free of water molecules and has only dry air in its pores. On being exposed to environment of lower humidity, rapid surface adsorption of water vapours into the porous film begins, causing rapid modulation of the light reflected from the prism base, the sensing area. This modulation is due to the appreciable changes in the r.i. of the film as RH increases. The graphs plotted for reflected intensity I_r of light against RH broadly fall

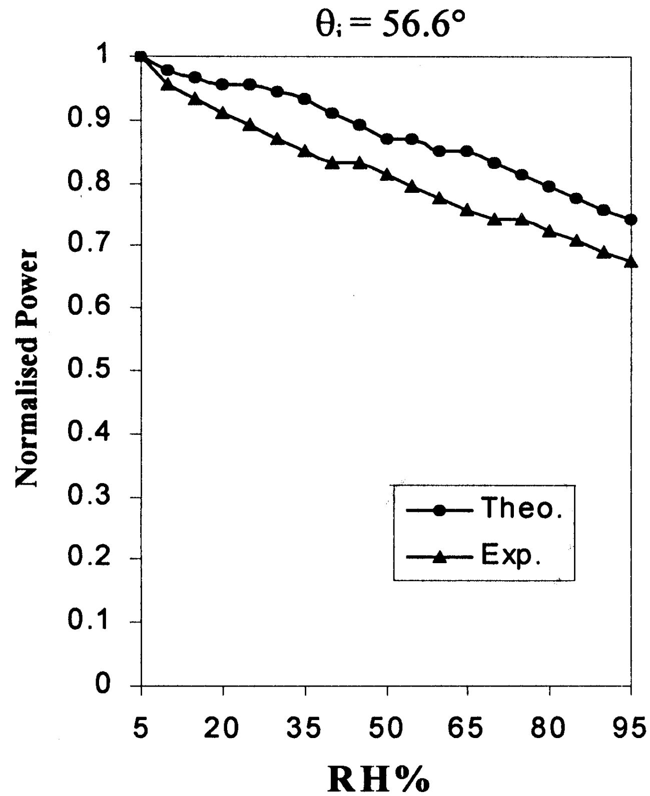

Fig. 9—Variations in the reflected intensity of light I_r against RH% with one layer of TiO_2 deposited at θ_i = 56.6° theoretical as well as experimental

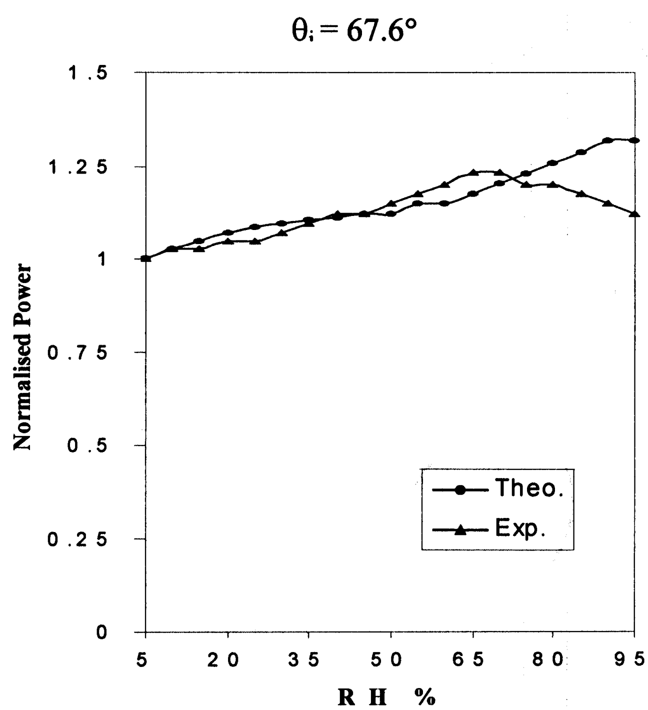

Fig. 10—Variations in the reflected intensity of light I_r against RH% with one layer of TiO_2 deposited at θ_i = 67.6° theoretical as well as experimental

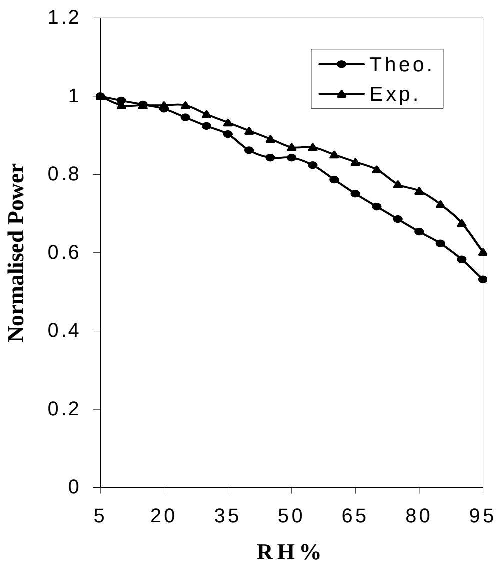

Fig. 11—Variations in the reflected intensity of light I_r against RH% with two layers of TiO_2 deposited at $\theta_i = 56.6°$ theoretical as well as experimental

under three categories: Type (a) I_r is falling continuously as RH increases. Curve a of Figs 7 and 8, corresponding to hole no.3 and $\theta_i = 56.6°$ showing continuous decrease in I_r as RH increases from 5% to 95% are of this Type. Type (b) I_r is rising continuously as RH increases. Curve c of Fig. 7 (corresponding to hole no.8 and $\theta_i = 67.6°$) and curve d of Fig. 8 (corresponding to hole no.11 and $\theta_i = 74.2°$) belong to this type. Type (c) I_r is showing rise and fall with RH in an irregular manner. Remaining graphs b, d and e of Fig. 7 and b, c and e of Fig. 8 fall in this category.

Variations in I_r falling under the categories (a) and (b) fulfil best the conditions required for the fabrication of a TiO_2 film based humidity sensor. The reasons for obtaining these three types of variations in reflected light intensity I_r can be explained in terms of four types of interfaces[16] created in the sensor configuration. These are (i) glass-TiO_2 film interface, (ii) glass interface with the film-moisture composite, (iii) TiO_2 film-moisture interface and (iv) glass-moisture interface. The r.i. of glass is 1.522 in the present case, that of moisture ranges from 1.00 to 1.33 and that of TiO_2 film lies between 1.8 and 2.1. Fresnel reflection taking place from these interfaces can be described on the basis of the nature of I_r-μ_a curve drawn in Fig. 1. It should be noted that the light reflected back from interface (iii) re-enters the glass through film-glass interface, interface (i), reinforces the light reflected from the other three interfaces.

It is thus seen that whereas the reflected intensity of light I_r from the interface (ii) increases, that from the interfaces (iii) and (iv) decrease, with increase in humidity. The light reflected from the interface (iii) re-entering the glass prism is determined by the interface(i) which lies between the glass and this interface(iii).

Hence, it is concluded that when reflections from the interfaces (iii) and (iv) are larger than those from (i) and (ii) over the entire humidity range, the total I_r due to all the four decreases with increase in humidity [categorized as reflection of type(a)]. On the other hand, if the reflection from the interface (ii) is larger than that from interfaces (iii) and (iv) over the entire humidity range, the total I_r increases with increase in humidity [categorized as reflection of type (b)].

Depending upon the angle of incidence and the consequent role played by reflection from the interfaces, there can appear also the irregular behaviour of I_r [categorized as reflection of type (a)]. Under this category I_r decreases in some parts of the graph and increases in the other parts of the graph, with increase in RH or vice-versa, giving rise to the irregular variations in I_r against RH. The same sensing elements were studied again after a lapse of five months to study the effect of aging. No aging effect was noticed and the results obtained earlier were reproduced.

It is important to note that the conical beam used as incident light in the experiment can also be produced with the help of an optical fibre. This would lead to a small sized compact humidity sensing device.

6 Conclusion

Results described clearly show that the prism based Opto-Electronic Humidity Sensors using titania films can be used with a high degree of accuracy to measure RH in the broad range of 5% to 95%. Of all the configurations, the highest sensitivity for entire range of humidity (~0.58) occurs for one layered film at $\theta_i = 56.6°$ corresponding to hole no.3. These sensors are reliable and cost-effective and can also be used at unmanned stations.

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