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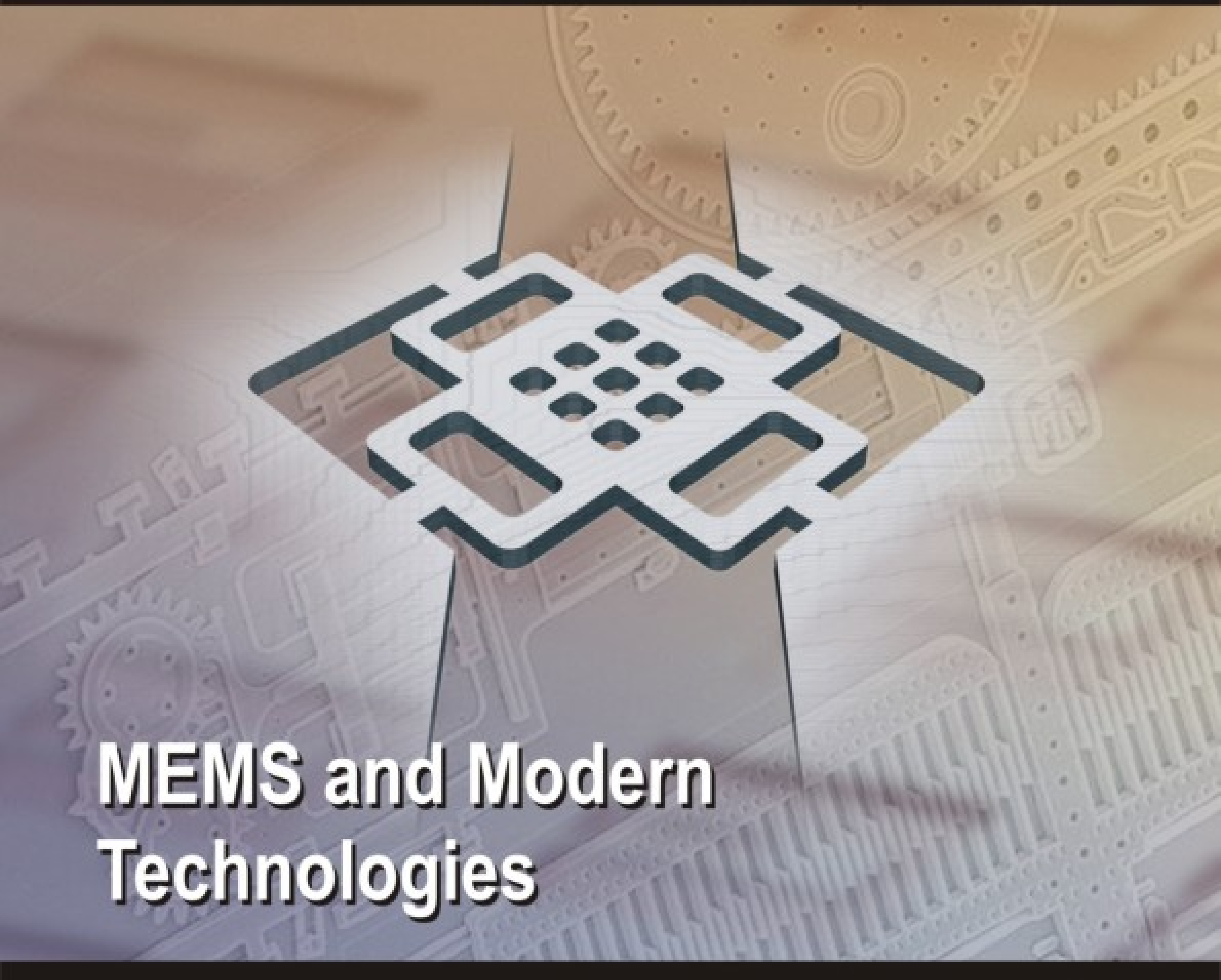

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Study of Optical Humidity Sensing Properties of TiO_2 and MgO Films

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Abstract: Paper reports a comparative study of humidity sensing properties of TiO_2 and MgO films fabricated by Sol-gel technique using optical method. One sensing element of the optical humidity sensor presented here consists of rutile structured two-layered TiO_2 thin film deposited on the base of an isosceles glass prism. The other sensing element consists of a film of MgO deposited by same technique on base of the prism. Light from He-Ne laser enters prism from one of refracting faces of the prism and gets reflected from the glass-film interface, before emerging out from its other isosceles face. This emergent beam is allowed to pass through an optical fiber. Light coming out from the optical fiber is measured with an optical power meter. Variations in the intensity of light caused by changes in humidity lying in the range 5%RH to 95%RH have been recorded. MgO film shows better sensitivity than TiO_2 film. *Copyright © 2007 IFSA.*

Keywords: Sol-gel process, Humidity sensors, Fresnel equations, Thin Films

1. Introduction

Prism based opto-electronic refractometers have been investigated by Bali et. al. [1-2] and K. Spenner et al. [3]. These refractometers are characterized by high sensitivities over a wide range of refractive index of the ambient. Base surface of the prism used in these refractometers is interfaced with the ambient. Out of several humidity sensitive compounds like inorganic acetates, halides, nitrates, sulphates, carbonates, phosphates and oxides, metal oxides are considered to be the most promising candidates for study because of their inherent chemical and physical stability [4-9]. Various oxides have been used as sensing elements for humidity and gaseous applications. Semi conducting oxides are known for their n-type conduction due to the presence of oxygen vacancies. Therefore, they prove to be good sensors for humidity. Earlier investigations based on the study of electrical behavior of thin films have, however, shown that among metal oxides, titanium dioxide is one of the most promising material for use in humidity sensors [10-14]. TiO_2 has a high capacity for adsorption of water molecules and its porosity is easily controlled [15]. Its anatase structure transforms into a more stable rutile structure at appropriate annealing temperatures [16].

The oxide mixtures of magnesium aluminate and magnesium ferrate have been used as humidity sensing elements [17]. MgO has been widely studied for its absorption properties for catalytic applications because of rich surface reactivities [18]. Moreover, MgO is thermodynamically stable and it is a material with wide band gap. It has application as a buffer layer in super conducting [19] and ferroelectric materials [20]. This material finds its use as secondary emission material for plasma display panels [21]. Due to high absorptive properties of this material, it also attracts its use as humidity sensor.

2. Principle of Operation

Consider an unpolarised beam of light of intensity I_i traveling through a medium of refractive index μ_g incident at an angle θ_i on the interface of this dielectric medium of refractive index μ_a. If θ_t is the corresponding angle of refraction, the intensity I_r of the light reflected back from the interface to the medium of r.i. μ_g, as obtained from Fresnel equations and Snell's law $\mu_g \sin\theta_i = \mu_a \sin\theta_t$ is expressed as [1]

$$\frac{I_r}{I_i} = \frac{1}{2}\left[\frac{\sin^2(\theta_i - \theta_t)}{\sin^2(\theta_i + \theta_t)} + \frac{\tan^2(\theta_i - \theta_t)}{\tan^2(\theta_i + \theta_t)}\right] \quad (1)$$

I_r/I_i plotted against μ_a for a given θ_i =55.6° and μ_g =1.522, yields a characteristic curve as shown in Fig.1. When $\mu_a < \mu_{ac}$ (i.e. upto point a) the incident ray undergoes total internal reflection (TIR). As μ_a increases and gets larger than μ_{ac}, I_r/I_i decreases; sharply at first (a to b), slowly afterwards, becoming zero at $\mu_a = \mu_g$ (point c). I_r/I_i starts increasing as μ_a increases beyond μ_g (c to d). If the glass surface of the base of the prism is interfaced with a TiO_2 film, then the glass-film interface behaves like a humidity sensor. Humidity detection would depend significantly on the thickness [1], of the deposited film, as the extent of absorption of water molecules depends on it.

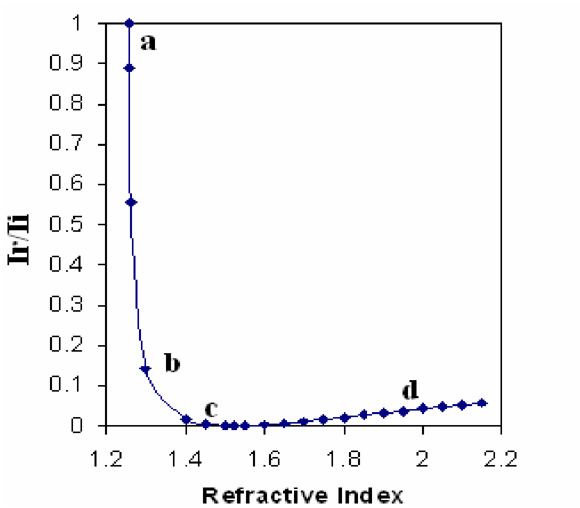

Fig.1. Characteristic curve showing variations in the ratio I_r/I_i with those in μ_a at an angle of incidence θ_i =55.6° and μ_g =1.522.

Sensitivity of sensor element is defined as the slope of the curve i.e.

$$Sensitivity = \frac{\text{Change in reflected output power}}{\text{Corresponding change in RH\%}}$$

$$S = \frac{\Delta I_r}{\Delta RH} \qquad \mu W / RH \text{ ,}$$

where RH (=Absolute Humidity/Saturation Humidity) is a dimensionless quantity [19]. It is worthwhile to mention that the sensitivity in the present paper has been calculated at different RH% 5, 15, 25, 35, 45, 55, 65, 75, 85 and 95%. Then average of all points is taken out.

3. Adsorption and Desorption Mechanism of Water Molecule

Fig.2 depicts this mechanism. Since water is a polar molecule, negatively charged oxygen of water molecule is electrostatically attracted to positively charged cationic side of metal oxide surface. If charge density of cationic side is low then water remains physically adsorbed at the surface by weak electrostatic field. When cationic charge density is high, as in the case of alkali salts, electrostatic force is high enough to form chemical bond between hydrogen and oxygen of water molecule, which in turn may break the bond between oxygen and one of the hydrogen atoms [20-22]. Mostly the force is high enough to break the bond in the initially adsorbed water vapor layer. Therefore, initial monolayer is generally chemisorbed. This chemisorbed layer can be removed thermally by increasing ambient temperature. Irreversible reaction for the first layer can be given as:

$$M^{+} \quad + \quad H_2O \rightarrow M\text{-}OH \quad + \quad H^{+}$$

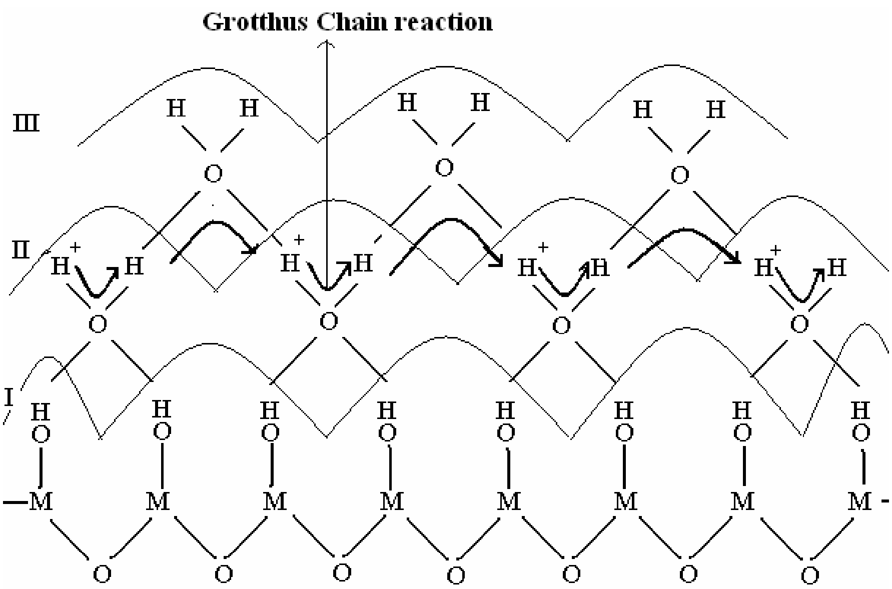

Fig.2. Simplified physical model of water adsorption proton charge conduction on the surface of a solid humidity sensor.

The complete water adsorption process is schematically depicted in fig.1. Subsequent water layers are physically adsorbed (shown as layer II in figure) on the first chemisorbed layer. This physisorbed water layer is bound by weak electrostatic force (known as hydrogen bonding) on the underlying chemisobed layer and can be reversibly removed by decreasing humidity. Therefore, this layer is mostly contributing to the humidity sensitive conduction of ceramic materials. The chemisorbed water molecules exert electrostatic field, which not only attract water molecules but also weaken oxygen to hydrogen bonds of physisorbed water molecules. Both, Frioiat [23] and Anderson [24], have concluded that weakening action of the surface electrostatic fields promotes dissociation of physisorbed water molecule in the following manner:

$$2H_2O \leftrightarrow H_3O^+ + OH$$

In pure water, the fraction of dissociated water molecule is approximately $1x10^{-8}$, but in the physisorbed layer of the water molecules this fraction is approximately estimated to be 0.01.

4. Sensor Fabrication

(a) Fabrication of TiO_2 Film

An isosceles prism of base angles 67.6° and refractive index 1.522 is used here. Prism base to be used as substrate is cleaned with the help of an ultrasonic cleaner (Model No. EN15U-S) supplied by Enertech Electronics Pvt. Ltd., India. Thin film of TiO_2 is deposited on the base of the prism with the help of sol-gel spin process. Solution used for this purpose is prepared by mixing isopropyl titanate and isopropyl alcohol in the ratio of 2:25 and deposition is done by setting the timer for 30 seconds and rotator for 3K rpm, which yields a film of approximate thickness of 1000 Å. After deposition, this film is dried for half an hour and then annealed at 450°C for four hours to make it rutile, porous and consequently sensitive to humidity.

(b) Fabrication of MgO Film

Magnesium metal is dissolved in anhydrous ethanol by refluxing for nearly 48 hours at boiling temperature of the solvent under anhydrous conditions. Once the metal is completely dissolved, reaction mixture is cooled to room temperature and filtered through sintered funnel in Nitrogen atmosphere. Thus, from filtrate, excess of ethanol is removed under reduced pressure leaving concentrated magnesium ethoxide in solution. At this stage a requisite composition of acetic acid and aqueous ethanol is added and resulting mixture is subjected to stringing for 24 hours at 85°C where ethoxy groups of magnesium ethoxide eventually gets substituted with acetate ones giving way to magnesium acetate in solution.

$$Mg(OC_2H_5)_2 + 2\ CH_3COOH \rightarrow Mg\ (OOCCH_3)_2 + C_2H_5\ OH$$

This is prepared precursor solution. It is found to be stable against hydrolysis for several weeks even in the most humid conditions in airtight container. Using this solution and following the fabrication method as for TiO_2, two-layered film of MgO is fabricated on the base of prism, dried for 30 minutes and then annealed at 450°C for four hours.

5. Experimental Setup and Working

Fig.3 describes experimental setup [22] and Fig.4 gives the ray diagram of the reflection through the film on the base of the prism. Prism is fixed in a rectangular cut on the wall of a specially designed steel chamber with the support of a metallic plate such that only base of the prism on which film is deposited remains inside the chamber. Light from an unpolarised 2 mW He-Ne Laser of wavelength 630 nm (OSAW, India) enters prism from one of its isosceles faces and gets reflected from glass-film interface at its base. Reflected light emerging out from other isosceles face of the prism is collected through an optical fiber and fed into an optical power meter (BENCHMARK, Model:FOPM-101) for measurement. Potassium sulphate is used as humidifier of the chamber and potassium hydroxide as dehumidifier. The humidifier /dehumidifier is kept in a dish over a stand. A thermometer having accuracy ±1°C is allowed to hang in the chamber. As relative humidity inside the chamber slowly increases intensity of reflected light I_r from the glass-film interface is recorded. This is done up to the value of relative humidity of 95%. In the process, temperature of the chamber decreases by 4°C. Chamber is then dehumidified first up to 10% RH by using dehumidifier potassium hydroxide and then up to 5% RH by carrying out heat cleaning cycle of the sensing element. The least count of hygrometer used here is 1% RH and that of optical power meter 0.1 dBm.

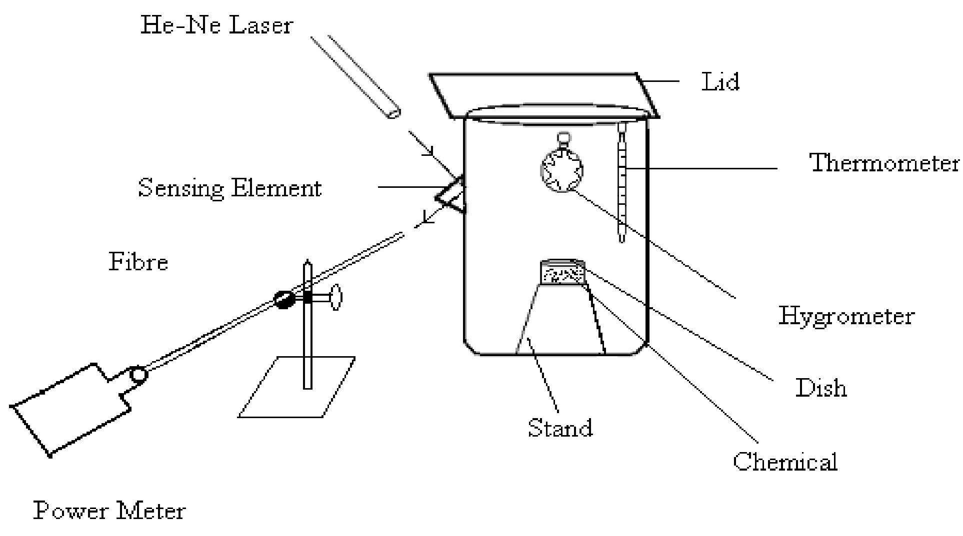

Fig.3. Experimental Set-up.

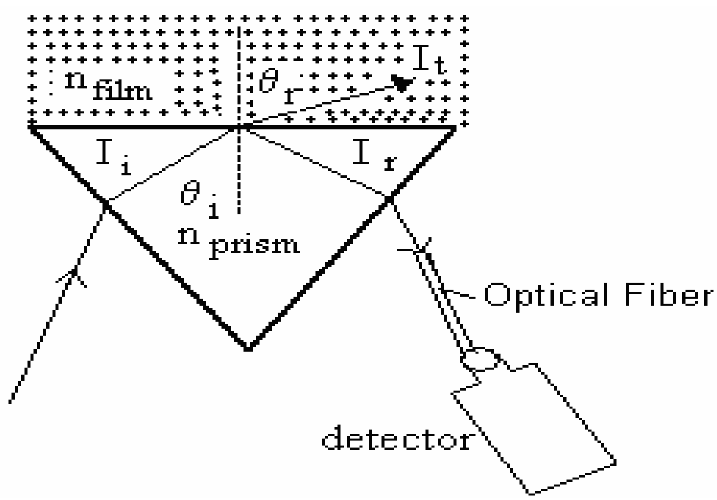

Fig.4. Ray Diagram of reflection through film on the base of the prism.

Temperature inside the chamber was 30°C in the beginning of experiment and it decreased by 4°C at the end of experiment when RH reached its maximum value 95%. Morphology of the film changes only when it is annealed at high temperature in the range 350°C-500°C. Change in output power due to small changes in temperature of 4°C is insignificant.

Angle of incidence of the light beam on the isosceles face of the prism is varied from 49.6° to 0.0° in such a way that the angle of incidence at the prism base varies from 37.6° to 67.6° in steps of 3.0°. As RH increases from 5% to 95% variation in reflected light intensity I_r has been recorded for each of these angles of incidence at the base surface of the prism.

6. Results and Discussion

Fig. 5 shows graph between normalized output power and %RH. Results are found to be reproducible. We have chosen one representative graph of each configuration in which normalized power has been plotted against change in RH%. It is observed that normalized power falls sharply with increase in RH%. Increase in humidity increases adsorption of water vapor and their condensation in pores of the film, causing a corresponding increase in refractive index of the film. This leads to a greater leakage [14] of light through the sensing element. Thus, intensity of light registered by power meter decreases with increase in humidity. Sensitivity of both sensing elements consisting of TiO_2 film and MgO film is measured by slope of curves 'a' & 'b' respectively shown in fig. 5.

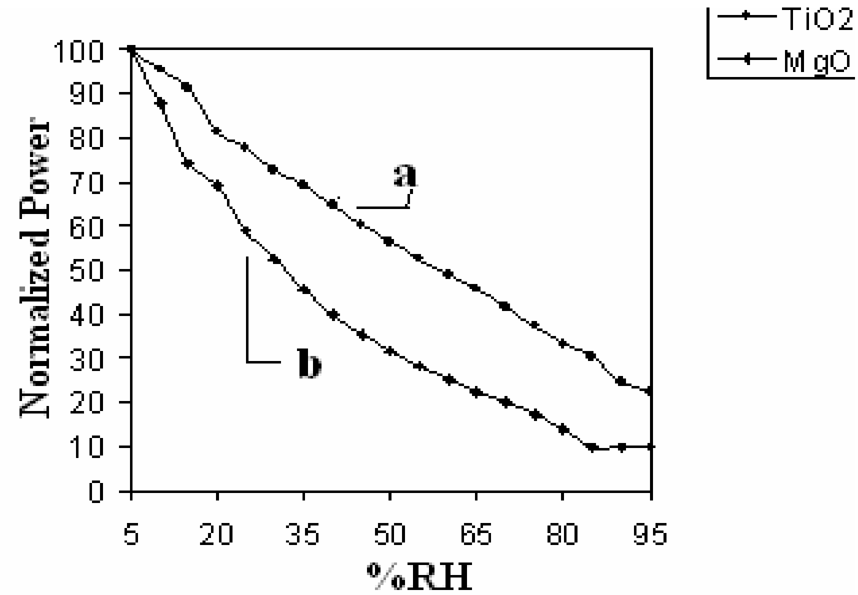

Fig.5. Variations in the reflected intensity of light I_r against %RH with two-leyered film of TiO_2 and MgO deposited on the base of prism at θ_i =55.6°.

It is seen that reflected output power at angle of incidence θ_i =55.6° decreases with increase in humidity in both cases (curves 'a' & 'b' fig.5). For the same initial incident power, reflected power is more than twice in the case of MgO film than that in the case of TiO_2 film. This is due to higher refractive index of TiO_2 film (~1.82) than the MgO (~1.70) film. Configuration with TiO_2 shows continuous decrease in output power throughout humidity range before saturation (curve 'a' Fig.5). Configuration with MgO shows high initial reflected power (Curve 'b' Fig.5). This output power decreases sharply in lower and middle humidity range i.e. from 5% to 80% but after 80%RH film becomes saturated and there is no change in output power. It shows very good sensitivity in range of 5% to 80%. The shape of these curves is analogous to the portion 'abc' of the theoretically determined curve shown in Fig.1. It is remarkable that when same sensing elements were studied again after a lapse of six months to study aging, no aging effect was noticed and results obtained earlier are reproducible.

7. Conclusion

Results show that humidity sensor with MgO film is more sensitive in the range of 5% to 80% than the sensor with TiO_2, while sensor with TiO_2 film is sensitive also in the higher range of humidity i.e. from 80% to 95%. Average sensitivity for the whole range of sensitivity viz. 5% to 95%RH in the case of MgO is found to be 1.00(μW/%RH) while in the case of TiO_2 this average sensitivity is 0.86(μW/%RH). Reliability and cost effectiveness are the characteristics of this sensor.

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Guide for Contributors

Aims and Scope

Sensors & Transducers Journal (ISSN 1726- 5479) provides an advanced forum for the science and technology of physical, chemical sensors and biosensors. It publishes state-of-the-art reviews, regular research and application specific papers, short notes, letters to Editor and sensors related books reviews as well as academic, practical and commercial information of interest to its readership. Because it is an open access, peer review international journal, papers rapidly published in *Sensors & Transducers Journal* will receive a very high publicity. The journal is published monthly as twelve issues per annual by International Frequency Association (IFSA). In additional, some special sponsored and conference issues published annually.

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Contributions are invited on all aspects of research, development and application of the science and technology of sensors, transducers and sensor instrumentations. Topics include, but are not restricted to:

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- Digital, frequency, period, duty-cycle, time interval, PWM, pulse number output sensors and transducers;
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- Technologies and materials;
- Nanosensors;
- Microsystems;
- Applications.

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