### Ag-polyaniline nanocomposite cladded planar optical waveguide based humidity sensor

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Received: 29 March 2008/Accepted: 24 July 2008/Published online: 14 August 2008 © Springer Science+Business Media, LLC 2008

Abstract The paper reports synthesis of Ag-polyaniline nanocomposite and its evaluation as an active optical cladding on a planar optical waveguide (POW) as a humidity sensor with variable concentration of the composite. The nanocomposite is dispersed in acetonitrile, and spin coated on the planar waveguide to form a clad. The system shows response to humidity, when tested in the range of 20-92% relative humidity (RH). A prism film coupling is used to characterize the cladded waveguide. The maximum sensitivity is seen for 47% concentration of the nanocomposite in acetonitrile. The response and recovery of the sensor are 8 and 55 s respectively. The low hysteresis is exhibited by the sensor. The sensor has repeatability and reproducibility. Material characterization is done using Scanning Electron Micrograph (SEM), X-Ray Diffraction (XRD), Ultraviolet Spectroscopy (UV) and Fourier Transform Infrared Spectroscopy (FTIR).

#### 1 Introduction

Planar optical waveguide (POW) sensors offer advantages like robustness, potential for integration with other optoelectronic components for compactness, and relative ease in applying specific coatings on the waveguide surface for evanescent wave sensing. The chemical/biological sensing

M. Kulkarni · B. B. Kale Center for Materials for Electronics Technology, Pashan, Pune 411008, India applications of integrated optical techniques have been a subject of various research undertakings. The optical sensing techniques have several advantages over electrical methods such as high sensitivity, immunity to electromagnetic interference and safety in detection of combustible and explosive materials.

POW formed by ion exchange on the surface of soda glass is sensitive to changes in parameters such as refractive index, light absorption and emitting processes such as chemiluminescence or fluorescence. The changes in the parameter cause modulation of light traveling within a glass waveguide and are useful for optical sensors in industrial and clinical applications. Deposition of clad of appropriate material allows the design of "all optical" device structures which must be capable of surviving under harsh working conditions for both civilian and military applications. Modern optical sensors allow rapid detection of minute changes in the refractive index of a thin film or the adsorption of chemical species from an aqueous solution. Such sensors are highly desirable in the chemical industry [1].

A typical planar optical waveguide (POW) consists of a substrate and a thin layer on the top (waveguiding layer) with refractive index higher than that of the substrate. Onto this, a sensitive covering material (clad) is deposited to detect an analyte. An interesting feature of such waveguides is that the electric field associated with a lightwave propagating in the waveguide layer is very strong at the surface of the POW. Highly sensitive optical monitoring can be performed for chemical species located at the POW surface on the basis of absorption and scattering of the guided light. Due to these reasons, evanescent wave techniques have been widely used to perform sensing at surfaces [2]. POW is also used because of its high sensitivity. To provide high sensitivity, the waveguide is made

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thin enough to support only a single mode and is fabricated from high refractive index materials. The sensitivity is lower for higher-order modes, so these are not often used in sensor applications.

Thin film POW sensors for gaseous analytes based on porous glass materials, prepared by the sol gel process are reported [3–5]. Ion selective and selective thin film (polymer) waveguide sensors are also reported [6, 7]. A polymer waveguide sensor with symmetric multilayer configuration was fabricated and its application in sensing low humidity concentration has been demonstrated by Ren et al. [8].

Different electrical and optical humidity sensors for such applications have been reported in the literature [9-12]. Planar Optical waveguide with screen printed thick films of semi conducting oxides is reported by Ansari et al. [13]. An evanescent-wave optical fibre humidity sensor using CoCl<sub>2</sub> doped thin polymer film coated on the bare fibre core is reported by Sunil et al. [14]. Nano- like Magnesium oxide films were studied as optical fibre humidity sensor by Shukla et al. [15]. A highly sensitive optical humidity probe based on reflectance measurements has been made using Nafion(R)- crystal violet (CV) films, which can be used to calibrate relative humidity in the range of 0-0.25% with a detection limit of 0.018%RH and exhibits low hysteresis [16]. The conventional materials used for sensing the humidity are electrolytic metal oxides, alumina thin films, and ceramics [17]. Porous ceramics or polymer films are used in commercially available humidity sensors. Presently polymers are identified as better candidates for practical applications; since they are compatible to oxides and ceramics and also they can be used at room temperature [18, 19] The humidity sensing using nanocomposites have been reported in the literature [20, 21]. Nanocrystalline materials have also been a subject of intense research in the recent years due to the unusual properties exhibited by them and also new physics generated by these systems. Das et al. have reported synthesis of nanocrystalline nickel oxide by controlled oxidation of Nickel nanoparticles and their humidity sensing properties [20], whereas the possibilities of using nanocomposites containing silver particles grown within a glass barium titanate composite by the ion exchange followed by reduction technique, for humidity sensing have been explored Pal et al. [21]. Humidity monitoring sensor with fast response-less than a couple seconds and high sensitivity based on the leaky plastic optical fibre structure was fabricated [22] Plastic optical fiber based humidity sensor with the Co-polyaniline nanocomposites clad was reported by Vijayan et al. [23].

Here, a POW based relative humidity sensor having an optical clad of Ag-polyaniline is evaluated in the range of 20–90%RH.

#### 2 Experimentation

#### 2.1 Fabrication of optical waveguide

A planar waveguide is fabricated on a soda lime glass substrate (size =  $25 \times 75$  mm and thickness = 0.8 mm, Refractive index (RI) = 1.515) using an ion exchange process [24, 25]. The substrate containing SiO<sub>2</sub>, Na<sub>2</sub>O, CaO etc. is kept in molten KNO<sub>3</sub> salt at 375 °C for 2 h in an alumina crucible, to achieve an optimized thickness of an ion exchange layer for single mode excitation (the diffused profile of  $K^+$  ions). The higher atomic weight  $K^+$  ions in the molten salt are exchanged with lighter Na<sup>+</sup> ions in the glass increasing the surface layer RI by 1.55 offering the waveguide characteristics to the substrate. The thickness of the waveguide, estimated using prismfilm coupling method, is 5 µm with 2 h of ion exchange treatment. The thickness of the ion exchange layer is negligibly small in comparison with the thickness of the substrate [26]. The diffusion constant for single mode optical guide in the exchanged layer was calculated from the equation.

$$d = 2(Dt)^{1/2}$$
(1)

where  $D = 1.08 \times 10^{-15} \text{ m}^2/\text{s}$  is the diffusion constant of K<sup>+</sup> ions in soda glass. The increase in the refractive index of surface layer (K<sup>+</sup>) than that of the substrate is about 1.5%. This increase in index exhibits light guiding property to the substrate. The waveguide thus prepared were characterized for their single mode operation as shown in Fig. 1a.

#### 2.2 Preparation of silver-polyaniline nanocomposite

All the chemicals and monomer used were of AR grade and is used as received. The solutions were prepared using doubly distilled water. The polymerization was initiated by the drop wise addition of the oxidizing agent,  $(NH_4)_2S_2O_8$ (dissolved in minimum amount of water) in an acidified solution of monomer containing synthesized silver nanoparticles by well known electrochemical method [27] under constant stirring at 0-5 °C. Para toluene sulphonic acid (p-TSA) was used as a dopant during in situ polymerization. The monomer to oxidizing agent ratio was kept as 1:1. After complete addition of the oxidizing agent, the reaction mixture was capped by stopper and kept under stirring for 24 h. The greenish black precipitate of the polymer was isolated by filtration and conditioned by washing and drying in an oven at 60 °C for 12 h. The solution so formed was further used for experimentation. Thus, this solution when used to deposit an optical clad is called as 100% concentration.



Fig. 1 (a) Experimental setup of sensor characterization system. (b) Single mode m-line pattern for waveguide kept for the ion-exchange treatment time of 2 h

# 2.3 Deposition of active clad of different concentrations

To get different concentrations (64%, 47%, and 37%), 0.1 cc of Ag-polyaniline nanocomposite was dispersed in 0.5, 1 and 1.5 cc of acetonitrile respectively. 0.1 cc of solution of various concentrations was spin coated on an optical waveguide to get an active clad for humidity sensing and the concentration is optimized.

0.1 cc of the optimized concentration of Ag-polyaniline nanocomposite was spin coated on single mode waveguide to get various clad widths (1, 2, 3, 4 and 5 mm) and then tested for RH response. The thickness of the deposited clads were measured by using Light Section Microscope (BK 70 × 50, VBE Carl Zeisco Jena, Germany). The minimum deposited clad thickness is higher than the penetration depth i.e. the evanescent field of light ( $\lambda = 632.8$  nm) in it [28]. The penetration depth ( $d_{clad}$ ) is calculated from [29]:

$$d_{\rm clad} = \left\{ k_0 \left( n_{\rm eff}^2 - n_{\rm clad}^2 \right)^{1/2} \right\}^{-1}$$
(2)

where  $n_{\rm clad} \& n_{\rm eff}$  are refractive index of clad and effective refractive index of guided mode respectively.  $k_0 = 2\pi/\lambda$  is the propagation constant and  $\lambda$  is the wavelength of light in free space.

#### 2.4 Characterization of optical waveguide and humidity sensor

The experimental setup is as shown in Fig. 1b. The turn table is used as a rigid support to mount the prism-film coupling assembly. He -Ne laser of wavelength 632.8 nm is used as the light source. The prisms used have a base of 10 mm and RI of 1.7. The sensor is enclosed in a glass chamber (volume 1.6 L, static system). He -Ne laser beam (beam diameter 100  $\mu$ m, 0.5 mW,  $\lambda = 632.8$  nm) is launched in, at critical angle [26, 29] with the help of input lens and a silicon photovoltaic detector are used to measure the intensity of the transmitted (output) light using a Rishcom digital multimeter. The optical waveguides are first characterized for the measurement of the number of guided modes. The transmission loss due to the glass chamber and the prism coupling is also taken into account while taking the humidity measurements [25, 29].

The humidity in the chamber is controlled by injecting humid or dry air through a nozzle. Care is taken to keep the prisms unaffected. The change in the RH% is measured directly, using a precalibrated hygrometer (Vaisala Humidity & Temperature Indicator HMI 31). The transmitted output light intensity is measured with respect to relative humidity for various values of concentration and clad width. First, the concentration dependence was studied and then, the width of cladding was optimized.

The sensitivity is defined as the change in the transmitted output (mV) per unit change (mV) per unit change in RH% i.e.  $\delta$  (mV)/ $\delta$  (RH%). The response time is measured for the RH transition from low level to high level i.e. to air ambient. The converse is done for measuring the recovery time. The maximum difference in two outputs i.e. increasing and decreasing cycles of RH, at constant RH level is defined as hysteresis.

#### 2.5 Material characterization

X-Ray diffraction was performed with a Philips X-Ray diffractometer (CuK $\alpha$  as radiating wavelength). FTIR spectra of the samples were recorded on a Perkin Elmer-Spectrum 2000 spectrophotometer operated between 400 and 4000 cm<sup>-1</sup> in a KBr medium. The morphology of the composite in the form of film was investigated with an Analytical Scanning Electron Microscope JOEL JSM

6360A. UV–Vis spectrum of the nanocomposites in acetonitrile was recorded by using Perkin Elmer spectro-photometer in the range of 200–800 nm.

#### 3 Results and discussion

#### 3.1 Material characterization

#### 3.1.1 SEM of Ag-polyaniline composite

Figure 2 shows the SEM micrographs of the films of various concentrations. In Figs. 2a, b and c void size appears to be the almost same. The number of pores is increasing and their size appears to increase with decreasing concentration. Figure 2d shows agglomeration of particles with least number of pores.

#### 3.1.2 XRD Ag-polyaniline composite

Figure 3 shows the XRD of Ag-polyaniline nanocomposites. In Ag-polyaniline nanocomposite, intense peaks having  $2\theta$  values of  $26^\circ$ ,  $56^\circ$  correspond to the peaks for polyaniline. Especially the sharp peak at 26° reveals the crystalline nature of the composite resulted due to the sequential growth of polyaniline chain on the silver nano-particles [30].

The peak of silver sulphide is seen at  $2\theta$  values  $35.13^{\circ}$  with corresponding plane (022). Peaks corresponding to  $2\theta$  values  $38.2^{\circ}$  and  $44.2^{\circ}$  reveal the crystalline nature of silver in planes (111) and (200) respectively.  $53.9^{\circ}$ ,  $2\theta$  value gives corresponding peak of AgCl in (201) plane.

#### 3.1.3 UV-vis of Ag-polyaniline composite

Figure 4 show absorption spectra of silver polyaniline nanocomposite after synthesis. UV–vis absorption spectrum is found sensitive to the formation of silver nanoparticles because silver particles can show an intense absorption peak around 400 nm originating from the surface plasmon absorption of nanosized silver particles. In comparison with the absorption spectra of pure polyaniline (Fig. 5) [31] it is seen that the shoulder peak at 320 nm is suppressed and the peak of 420 nm is enhanced this may be due the presence of Ag nanoparticles in the nanocomposite.



Fig. 2 Scanning electron micrographs of films of various concentrations of Ag-polyaniline composite (from (a) 100%, (b) 64%, (c) 47% and (d) 37%)



Fig. 3 XRD of Ag-polyaniline nanocomposite



Fig. 4 UV-vis absorption spectra of Ag-polyaniline nanocomposite



Fig. 5 UV-vis absorption spectra of pure polyaniline

#### 3.1.4 FTIR of Ag-polyaniline composite

The peak observed (Fig. 6) at  $814.42 \text{ cm}^{-1}$  is the characteristic of paradistributed aromatic rings indicating

polymer formation. C–H in plane and C–H out of plane, bending vibrations appear at 1126.58 and 684.46 cm<sup>-1</sup>, respectively. Aromatic C–N stretching indicating appearance of the secondary aromatic amine group is observed at 1303.22 cm<sup>-1</sup>. Of particular interest are the bands in the vicinity of 1399.07 and 1588.16 cm<sup>-1</sup> corresponding to the benzoid ring and quinoid ring modes respectively. The

presence of these bands clearly shows that the polymer is composed of insulating and conducting phase [31, 32]. Band at 2928.28 cm<sup>-1</sup> is assigned to aromatic C–H stretching vibration.

Presence of all these characteristics bands confirms that the polymer is in the conducting ES phase. The metal oxygen stretching frequency of Ag–O is at 567  $cm^{-1}$ .

#### 3.2 Optical waveguide characterization

The K<sup>+</sup> ion exchanged waveguide, characterized using prism-film coupling has given a single mode (m = 0) excitation for soda glass. After arranging the waveguide setup for *m*- lines (m = 0), the insertion loss, as measured without cladding is 0.78 dB/5 cm, obtained from the intensity of the direct source (V1 = 325 mV) and that of the transmitted light through the waveguide without cladding (V2 = 300 mV). The guide thickness calculated by the diffusion equation gave a thickness of 5 µm for an ionexchange time of 2 h.

## 3.3 Humidity response for Ag-polyaniline nanocomposite

The effect of humidity on a planar uncladded waveguide is negligible within the accuracy of detector (Least count = 0.1 mV). The initial insertion loss is measured as the ratio of output of cladded guide to the intensity launched and is 1.16 dB/cm (12%). In general, there is decrease in light transmission through the guiding layer with increasing RH exhibiting two to three sensitivity regions over the tested range.

Figure 7 shows the calibration curve for humidity, for samples with different concentrations from 100% to 37%. The sensitivity decreases with increasing concentration. The humidity sensing is enhanced by the increase in pores with decrease in concentration but for 37% concentration there is an agglomeration of the nanocomposite due to which the sensitivity has further decreased. The concentration of 47% showed the maximum sensitivity. Thus, for the optimized concentration the response of the sensor for varying clad width to humidity were done.

Table 1 gives the region wise sensitivity of concentration variation with respect to relative humidity. The absorption of small amount of light energy is present at the clad air interface at lower RH (Region I  $\sim$  from 20 to



Fig. 6 FTIR spectra of Ag-polyaniline nanocomposite



**Fig. 7** Calibration curves of concentration variation (100–37%) of Ag-polyaniline nanocomposite with relative humidity

45%RH) as the surface adsorption of water vapors takes place dominantly. The clad thickness is quite large compared to guide thickness and also the wavelength of light used ( $\lambda = 632.8$  nm), and so the guide clad coupling is not much affected. This is the reason for getting less sensitivity at low RH.

In the intermediate RH range (Region II  $\sim$  from 45 to 80%RH), few layers of water molecules will adsorb on the walls of capillaries and in the empty pores in the clad. This

changes the RI of the clad, as the air is replaced by water vapor. This may cause large radiation in the clad [13] supporting the transverse guiding as it becomes higher index layer having lower index medium from both sides satisfying the guiding conditions.

At higher RH level, (Region III  $\sim$  from 80 to 92%RH) the water adsorption by the clad no longer remains multilayer adsorption. It results in total capillary condensation in the pores of the clad, tending towards saturation. The water molecules might be penetrating up to the clad guide interface causing the dominant change in RI depending on the porosity and pore size. In this situation, maximum loss of energy is expected into the clad. This loss may be the additional effect of radiation of light energy at the clad guide interface itself. Therefore all the energy of the evanescent field is lost at the clad guide interface itself reducing the confinement. This is the reason for reduction in sensitivity leading to the saturation at very high RH level.

Figure 8 shows the variation of output for various clad width from 1 to 5 mm for 100% concentration. It is observed that the sensitivity increases up to the clad width of 3 mm and then decreases with further increase of width. Optimized clad width (3 mm) exhibited best sensitivity and is somewhat linear. As the electromagnetic wave in the form of incident light guided propagates through the sensing region, the optical power in evanescent tail of the propagating mode is absorbed with the change in the environmental parameter i.e. humidity. This results in a

Table 1 Region wise sensitivity of concentration variation with respect to relative humidity

Conc. %	Thickness (µm)	Refractive index	Region 1 (mV/%RH)	Region 2 (mV/%RH)	Region 3 (mV/%RH)
100	20.68	1.447	35.2(42-20)	14.4(78–43)	12.2(92–79)
64	18.65	1.428	35.6(44-20)	20.2(76-45)	15.0(92-77)
47	16.56	1.422	39.1(40-20)	22.4(79-41)	18.9(92-80)
37	15.23	1.419	25.7(42-20)	16.5(75–43)	6.8(92–76)



Fig. 8 Calibration curves of variation of clad width (1–5 mm) of Agpolyaniline nanocomposite with respect to humidity

modulated output from the fiber and is used as the criterion for detecting and determining the relative humidity in the surrounding.

The thickness of the films used here are in the range of 15 to 20 µm i.e. about an order of magnitude higher than the basic guide thickness 2 µm and much higher than wavelength of light used (632.8 nm). The refractive indices (RI) of clads are less than guide RI as required for waveguides. These are higher than air RI. Therefore, they offer higher transmission loss and more leaky field in the clad [26]. The leaky field increases with increase in RI due to adsorbed moisture. The porous nature of the clad allows light to penetrate deeper inside the clad elongating evanescent field of guide into the clad. During this process some part of the light reflects back to the guide from clad guide interface or scatters from the pores of the clad and overlaps with evanescent field of guide at the clad-guide interface. This maintains the coupling between the guide and the clad [13]. It is reported that the porosity provides a large number of scattering centers which effectively acts as luminescence centers due to scattering and enhance the light intensity in the clad region in addition to the



Fig. 9 Comparison of pure polyaniline and Ag-polyaniline nanocomposite for humidity response

elongation of evanescent field [33–35]. The increase in clad width provides larger area for water adsorption increasing the sensitivity with length. Further, decrease in sensitivity for clad width greater than 3 mm may be due to the absorption of light by the material itself.

Ag-polyaniline nanocomposite is more sensitive to humidity in comparison to polyaniline alone (Fig. 9). This clearly shows that Ag nanoparticles play an important role in enhancing the humidity sensing.

5 cycle to cycle variations were studied for checking the repeatability (Fig. 10). 5 samples were checked for humidity response to study its reproducibility (Fig. 11). About 3% uncertainty from cycle to cycle and uncertainty of about 4% from sample to sample is observed, indicating better repeatability and reproducibility. The response and recovery of the sensor are 8 and 55 s respectively. Hysteresis is observed to be nearly 2% for all of the concentrations as seen from Fig. 12. The hysteresis at lower humidity is a result of slow desorption of the water from the pores of the capillary. At higher humidity, the capillary condensation occurs and forms a meniscus over the capillaries of the film, which attributes to hysteresis and nonlinearity in the response.



Fig. 10 Variation from cycle to cycle of a typical sample for 47% concentration and 3 mm clad width of Ag-polyaniline nanocomposite



Fig. 11 RH response of different samples for 47% concentration and 3 mm clad width of Ag-polyaniline nanocomposite

#### 4 Conclusions

An optical waveguide sensor has been proposed that is potentially suitable for detecting humidity over the range of 20–92%. Ag-polyaniline nanocomposite clad on a planar optical waveguide can serve as an excellent candidate for optical sensing. This sensor is repeatable, reproducible with fast response and recovery. The added advantages of the sensor are small size, less cost and easy fabrication.



Fig. 12 Variation of humidity for increasing and decreasing cycle of a typical sample for 47% concentration, 3 mm clad width of Agpolyaniline nanocomposite

**Acknowledgments** One of the authors, Madhavi Fuke would like to acknowledge Center for Sensor Studies, University of Pune, for their financial support.

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