Optical coatings on fiber for relative-humidity sensing applications

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Received January 4, 2013; accepted February 26, 2013; posted online June 14, 2013

A fiber-optic relative-humidity (RH) sensor composed of multilayer of porous dielectric films is proposed. The transducer deposited on fiber end-face is multilayer coating consisting of nano-porous TiO_2 and SiO_2 films, which forms a low-fineness Fabry-Perot (F-P) filter with one of minimum reflections at about 1 350-nm wavelength. The dielectric thin films realized by e-beam evaporation without ion-source assistance have columnar and porous structures, which exhibit sensitivity to RH change of environments. When the sensor is exposed to an environment of RH change from 10.9% to 92.8%, experimental results demonstrate 77.9-nm shift of characteristic wavelength.

OCIS codes: 310.1620, 310.1860, 310.4165, 310.6845.
doi: 10.3788/COL201311.S10404.

With the development of science and technology, humidity measurement is vital importance in numerous fields such as aerospace, electronic processing, food processing, air conditioning, storage, and pharmaceutical industry and agriculture[1−4]. Different from traditional capacitive and resistive humidity sensors which cannot keep long-term stability and interchangeability in the serious pollution and strong electromagnetic interference conditions, optical fiber humidity sensors have received much attention due to its intrinsic properties such as miniature, high sensitivity, fast response, and immune to electromagnetic interference.

In this letter, a fiber optic relative-humidity (RH) sensor composed of multilayer of porous dielectric films is proposed and demonstrated for RH measurement. The transducer deposited on fiber end-face is a three-layer membrane of TiO_2 / SiO_2 / TiO_2, which forms a low-fineness Fabry-Perot (F-P) filter. The dielectric thin films realized by e-beam evaporation without ion-source assistance have columnar and porous structures, which exhibit sensitivity to RH change of environments. The reversible absorption and desorption of water molecules in the porous films in dependence on water vapor shifts the reflected fringes of F-P filter, therefore humidity sensing is correlated with the shift of characteristic wavelength of the F-P sensing head.

The schematic diagram of miniature F-P RH sensing probe is presented in Fig. 1, which is formed by a single mode fiber (SMF: 9 /125 nm) and three layer optical films manufactured by e-beam evaporation.

As shown in Fig. 1, the combination of films can be considered as F-P interferometer, the two reflective mirrors of the hybrid F-P cavity are the interfaces of TiO_2 films. Only consider the interference caused by first reflected light, the optical path difference of a round-trip propagation is

$$\delta = 2nL + \frac{\lambda}{2}, \tag{1}$$

where n is the equivalent refractive index of the region between the two mirrors, L refers to the thickness of the three layers films, λ means the wavelength, and there is a 1/2 phase shift at each reflection from optically thinner medium to optically denser medium. Combined with Eq. (1), it is assumed that the wavelength of the resonance dip λ_k in the reflection spectrum satisfies

$$(2k+1) \times \frac{\lambda_k}{2} = 2nL + \frac{\lambda_k}{2}, k = 1, 2, 3 \cdots. \tag{2}$$

So the λ_k can be expressed by

$$\lambda_k = \frac{2nL}{k}. \tag{3}$$

When the sensitive probe is exposed to different humidity levels, the equivalent refractive index of the region will change, which will cause a drift of the resonance wavelength in the reflection spectra,

$$\Delta\lambda + \lambda_k = \frac{2(n+\Delta n)L}{k}. \tag{4}$$

As observed from Eq. (4), the wavelength shift is a function of equivalent refractive index. The relation between refractive index and humidity levels is depended on the coating process[5].

The proposed probe is an optical film at the fiber interface deposited by e-beam evaporation, which forms a F-P cavity. Referring to Fig. 2, the dielectric thin films realized by e-beam evaporation without ion-source assistance have three layers and porous structures, which exhibit sensitivity to RH change of environments. The

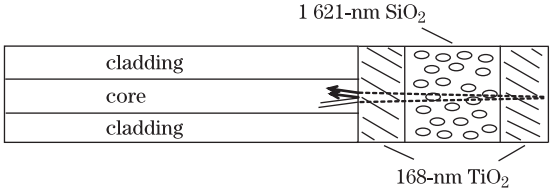

Fig. 1. Schematic diagram of the $TiO_2/SiO_2/TiO_2$ combination of films F-P RH sensor

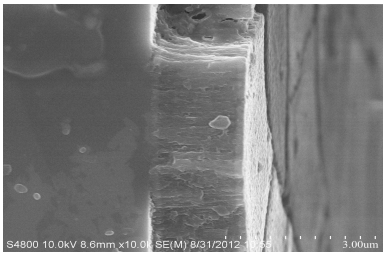

Fig. 2. Scanning electron microscope (SEM) image of the sensing probe.

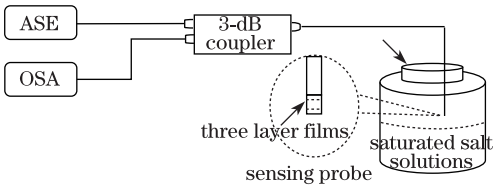

Fig. 3. Experimental set-up of the humidity measuring system.

evaporation coating process as follows: filling gas is oxygen (O_2) with a velocity of 100 sccm, baking temperature 100 °C and working air pressure 0.046 Pa. The first film closed to optical fiber section is TiO_2 film (168.55 nm) deposited under the e-beam current of 240 mA. The e-beam current of second film of SiO_2 (1 621.34 nm) is 44 mA, and the third film has the same coating process as the first TiO_2 thin film.

As shown in Fig. 3, the proposed fiber optic humidity sensing system consists of an amplified spontaneous emission (ASE) broadband light source (center wavelength: 1 550 nm, full–width of half–maximum (FWHM): 60 nm), optical signal analyzer (OSA) (YOKOGAWA, AQ-6370B, wavelength range: 600–1 700 nm), optic fiber coupler (OC) (a fused tapered optical fiber 50/50 coupler at 1 310 nm), and the F-P sensing probe[6,7]. Firstly, the light emitted from the ASE broadband light source is coupled to the OC with a standard SMF. One of the output SMF is connected to the OSA for measuring the reflected optical spectrum, while the second output is fusion spliced with the fabricated sensing probe.

The humidity sensor was enclosed in a sealed receptacle with a cylindrical foam-rubber cushion, it was suspended in the air above different saturated salt solutions to measure different values of RH. Table 1 shows the different values of RH in the air of the sealed receptacle for each saturated salt solution at 30 °C (room temperature), according to the salt solution saturated humidity value standard made by the International Organization of Legal Metrology. And the actual humidity value was measured by Center313 Hygrothermograph (rang: temperature –20–60 °C, humidity 0–100% RH; accuracy of measurement: temperature ±0.7 °C, humidity ±2.5 %RH) in the laboratory room (30 °C, 63.2–64.8 %RH).

All experiments were performed inside a clean room with stable temperature (30°C) and RH (62.7–64.2 %RH).

Considering that the balance of humidity in the containers would be damaged every time when the lid of the bottle is open to change the environment humidity container, 10 min was given to reach equilibrium at each humidity level before recording and the measuring sequence from small to large RH with the solutions of LiCl, $MgCl_2$, NaBr, NaCl, KCl, and K_2SO_4, respectively. Each group of data was re-measured in three weeks to verify its repeatability.

Figure 4 shows the final reflection spectra of the sensing probe after contracting the spectra of light source in different humidity levels of LiCl, $MgCl_2$, NaBr, NaCl, KCl, and K_2SO_4. With the environment humidity increased from 10.9% to 92.8%, the reflection spectrum curve was shifted towards the longer wavelengths (red shift) as far as 78 nm, Table 2 shows the evolution of spectra shift during the change of RH.

As shown in Fig. 4, the wavelength of minimum reflection shifts under different humidity levels. Wavelength drift demonstrates a significant phase shift of the interference fringes. The shift of the interference fringes indicates the refractive index change of optical film, which causes changes in optical path and therefore results in wavelength shifts in response to the optical path modulation.

Wavelength shifts of the sensing probe under different humidity levels in both ascending and descending order is shown in Fig. 5. The experimental results are close to each other when the RH was varied in ascending and

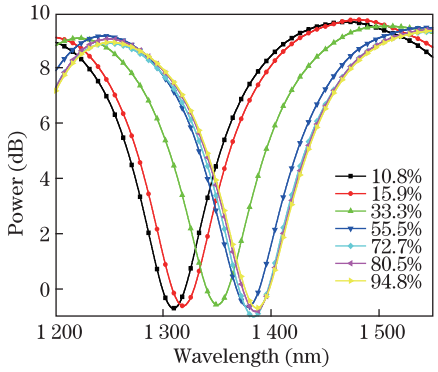

Fig. 4. (Color online) Wavelength shift of the porous films sensor in different environments of varying RH.

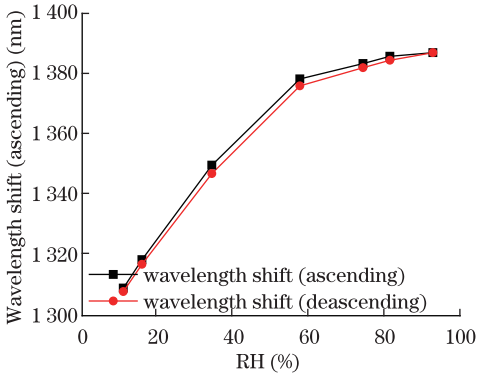

Fig. 5. (Color online) Wavelength shift in different humidities.

Table 1. Different Values of RH in Air of Sealed Receptacle for Each Saturated Salt Solution at 30 °C

Solutions	LiBr	LiCl	$MgCl_2$	NaBr	NaCl	KCl	K_2SO_4
Humidity	6.2±0.5%RH	11.3±0.3%RH	32.4±0.2%RH	56.0±0.4%RH	75.1±0.2%RH	83.6±0.3%RH	97.0±0.4%RH
Actual Humidity Value	10.9%RH	15.8%RH	34.3%RH	57.6%RH	74.3%RH	81.4%RH	92.8%RH

Table 2. Corresponding Wavelength Values of Troughs and its Variations in Different Humidity Environments

Solution	LiBr	LiCl	$MgCl_2$	NaBr	NaCl	KCl	K_2SO_4	Total Drift
Actual Humidity Value	10.9%RH	15.8%RH	34.3%RH	57.6%RH	74.3%RH	81.4%RH	92.8%RH	
Wavelength (nm)	1 309.1	1 318.5	1 349.8	1 378.3	1 383.4	1 385.8	1 387.0	
Drift (nm)		9.4	31.3	28.6	5.1	2.3	1.2	77.9

descending order. The RH sensitivity of the proposed sensor is 0.89 nm/%RH, which is higher than other resonance wavelength detection based fiber sensors[1]. Especially, in the low humidity range (10.9–57.6 %RH), the spectrum shift reaches 69.3 nm, which means RH sensitivity of 1.48 nm/%RH. The proposed sensor shows promising prospect in the low humidity measurement.

In conclusion, according to fiber F-P cavity theory, an optical fiber humidity sensor with multilayer film is proposed and manufactured by e-beam evaporation. The sensitivity is characterized by experiment, the wavelength shift has reached 77.9 nm when RH ranges from 10.9% to 92.8%. However, in the low humidity area (10%–57%), the measured RH sensitivity of the proposed sensor is determined to be 1.47 nm/%RH, which shows an excellent low moisture sensitivity. Further investigations will focus on the improvement of sensitivity in higher humidity and repeatability of the multilayer sensor. Also intensive study will be conducted on the correlation of different film structures with the RH sensitivity. In general, the combination of optical fiber sensor with thin film optics is very promising for application in humidity sensing.

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