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High-speed optical humidity sensors based on chiral sculptured thin films

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ABSTRACT

A high-speed humidity sensor based on a nanostructured chiral sculptured thin film (CSTF) was fabricated and tested. The sensing principle is based on the shift of the central wavelength of the circular Bragg regime of the CSTF caused by adsorption and desorption of water vapor in the void regions of the CSTF. Spectral changes due to varying relative-humidity (RH) levels in the ambient environment were measured by a spectrometer. The CSTF sensor exhibited excellent reversibility and reproducibility from 40% to 100% RH. Moreover, the adsorption time of the sensor was measured to be as low as ~140 ms, making it promising for many high-speed humidity-sensing applications.

1. Introduction

Humidity sensors have many different applications ranging from nuclear power reactors to residential air conditioners [1–4]. In recent decades, various sensing modalities have been developed, typically exploiting changes in resistance, capacitance, or refractive index (RI) to characterize changes in relative humidity (RH) [5–8]. The most widely used humidity-sensing materials include porous ceramics (*e.g.*, Al_2O_3 and TiO_2) [9–14], nanostructured metal oxides [15–17], polymers (*e.g.*, polyimide and phthalocyanine) [18–20], and polyelectrolytes (*e.g.*, sulfonated polysulfone and polyvinyl acetate) [21,22].

The criteria for a desirable humidity sensor include high sensitivity, long-term durability, fast response, low cost, and operation over a wide range of humidity and temperature. A particularly important parameter of humidity sensors is their response speed. A response time on the order of several seconds is considered high-speed for humidity sensors today [23,24]. However, humidity sensors with sub-second response times will be crucial for numerous applications including control of industrial processes, monitoring atmospheric RH, managing patients undergoing anesthesia, and pulmonary-function diagnostics [25,26]. Sub-second humidity sensors using inorganic materials have been demonstrated based on porous nanostructures with different materials and architectures [27–34]. Humidity sensors using organic materials have also been developed using photoresist and polyimide [27,35]. Humidity-sensing devices [36,37] using nanoporous polymer structures have been made by holographic photopolymerization [38,39]. Table 1 summarizes the typical response time of the reported high-speed humidity sensors. Among the aforementioned humidity sensors, metal oxide based sensors are promising in terms of their response speed, stability, and repeatability. Most of these sensors are capacitive, involving electrical measurements. Relatively few optical sensors have been demonstrated, and they are generally based on a narrow bandpass filter using TiO_2 nanostructures [40–42].

In this paper, we report the fabrication and characterization of an optical humidity sensor based on a chiral sculptured thin film (CSTF). The CSTFs consist of arrays of helical nanowires that are parallel and nominally identical to each other, providing the CSTF with a distinguished axis of chirality that is aligned normal to the substrate on which the CSTF is deposited [43]. This helical morphology gives rise to a resonance for either left- or right-circularly polarized (LCP or RCP) light, with the resonance regime being called the circular Bragg regime [43–45]. Most importantly, when the wave vector of the incident CP light is parallel to the axis of chirality and the CSTF is sufficiently thick, CP light of the same handedness as the CSTF is highly reflected, but CP light of the other handedness is only slightly reflected in the circular Bragg regime. A CSTF is thus a circular polarization filter [45]; this characteristic has also been exploited to realize CP light emission from light-emitting devices made of CSTFs [46,47]. The circular polarization-sensitive properties of a CSTF depend on

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Table 1
Reported adsorption and desorption times of various humidity sensors.

Material	Adsorption time	Desorption time	Reference no.
Porous silicon oxide	35 ms	25 ms	[27]
Nanoporous silicon thin film	200 ms	N.A.	[28]
Rhodium electroplated sensor	30–50 ms	30–50 ms	[29]
Nanostructured metal oxide	<220 ms	>400 ms	[30–33]
Polyimide columnar film	1 s	N.A.	[35]
Helical photoresist structure	75 ms	175 ms	[27]
Porous polymeric grating	137 ms	9 s	[36]
Porous polymeric photonic crystal	1.5 s	20–30 s	[37]

the material chosen to fabricate it, the deposition pressure and temperature, and most significantly, the direction of the adatom flux with respect to the substrate during deposition. Generally, smaller angles between the adatom-flux direction and the substrate correspond to more porous CSTFs [43]. To our knowledge, the circular Bragg regime has been experimentally validated for humidity sensing only by Lakhtakia et al. [44], that too only qualitatively.

The central wavelength and the full-width-at-half-maximum (FWHM) bandwidth of the circular Bragg regime are dependent on the permittivity mismatch between the helical nanowires and the void regions of the CSTF, in addition to its porosity [48,49]. Examination of scanning electron micrographs indicates that the void regions exist on two length scales: between the helical nanowires (submicron scale) and inside the nanowires (nanoscale) [43]. Upon exposure to varying RH, the water vapor diffuses in/out of the void regions, inducing changes in the overall dielectric properties of the CSTF, as well as the parameters of the circular Bragg regime. Theory predicts that the spectral shift of the circular Bragg regime can be used to quantitatively sense the RH change of the environment [48,49]. As we will demonstrate in Section 3, this inorganic-material-based humidity sensor offers high-speed sensing, long-term stability, and desirable reproducibility. Additionally, as electron-beam evaporation is a workhorse technique commonly used by manufacturers [43,50], this optical humidity sensor will also be inexpensive.

2. Description of experiments

CSTFs were deposited through an electron-beam evaporation system configured for the serial bideposition (SBD) technique [45,51], in which the substrate was tilted at an angle of 15° measured between the direction of the impinging vapor flux and the substrate plane; this angle is denoted by χ_v in Fig. 1. By periodically rotating the substrate 180° about the substrate normal (the z-axis in Fig. 1) during the deposition process, a CSTF with desired helical morphology can be obtained. The pitch of the helical nanowires in the CSTF is defined as the width of one complete turn along the axis. The void region between the helical nanowires and the volume fraction of the deposited material can also be experimentally controlled with this process.

The CSTFs were deposited onto flat 1 in. × 1 in. glass substrates (Corning 7059). The distance between the evaporation source and the center of the substrate was 10 in., and the deposition rate was controlled at 2.5 Å/s, which was monitored by a resonating quartz crystal sensor at normal incidence located near the substrate. A well-controlled deposition rate resulted in a uniform pitch. The vacuum base pressures were below 4×10^{-6} Torr. Titanium dioxide (TiO_2) was chosen as the material for deposition, because of its high bulk refractive index ($n_{TiO_2} = 2.6$) and excellent transparency at visible wavelengths. In addition, TiO_2 demonstrates the photocatalytic self-cleaning phenomenon: ultraviolet illumination of TiO_2 generates electron-hole pairs that react with organic contaminants on the surface to produce several compounds including CO_2 and H_2O [34,52], which desorb from the surface. This self-cleaning effect provides a solution to reduce the impact of sensor aging. Finally, the optical properties of TiO_2 do not change significantly over common environmental temperature ranges.

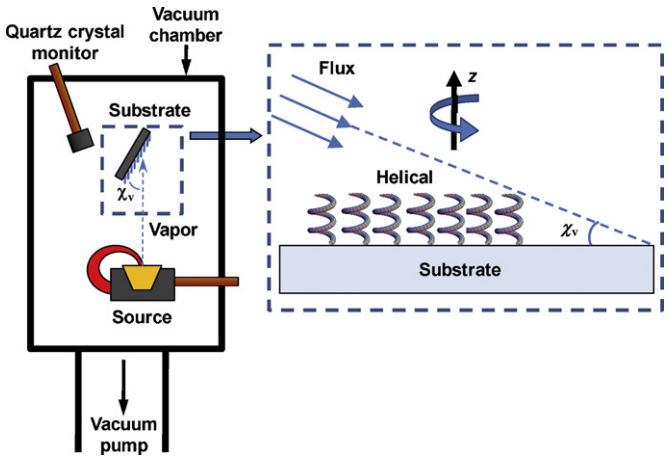

Fig. 1. Schematic of the oblique angle deposition process.

Our setup for the rapid optical sensing of humidity is illustrated in Fig. 2. A structurally right-handed CSTF was attached to a Teflon substrate. Light from a halogen lamp was made either LCP or RCP through a combination of a linear polarizer and a quarter-wave plate, and was then used to illuminate the CSTF normally. The total transmittance through the CSTF was measured over the 500–900 nm wavelength range with a spectrometer (Ocean Optics Co., HR4000). A humidity chamber (ESPEC North America Inc., SH-241) integrated with a water tank and a fan system was used to control the operating temperature and the RH. The RH inside the humidity chamber could be programmed and adjusted to any value between 40% and 95%.

However, because the humidity chamber required almost 15 min to adjust the RH, we were forced to use a separate method to characterize the sensor's response time. This was done using a small, transparent chamber (4 cm × 4 cm × 4 cm), where the RH can be made to quickly oscillate between 20% and 100%. RCP light from an external He–Ne laser diode ($\lambda = 633$ nm) was guided normally through the structurally right-handed CSTF, where the transmitted intensity was collected by a photodetector. Step-like intensity changes (corresponding to the transmittance changes in the circular Bragg regime of the CSTF induced by the RH oscillations within the chamber) were captured through the photodetector. The response time was then extracted from the recorded data.

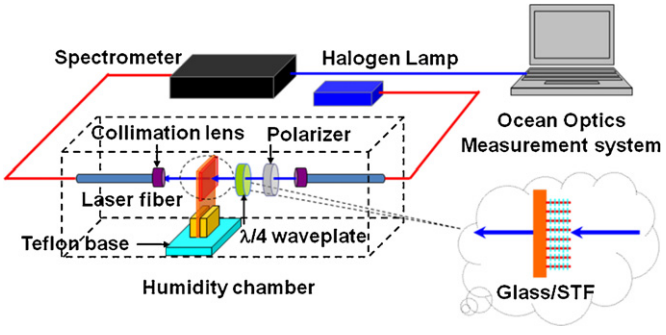

Fig. 2. Schematic of the humidity-sensing measurement setup.