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Research paper

Carbon-nanotube / Polyvinyl alcohol coated thin core fiber sensor for humidity measurement

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ABSTRACT

A Carbon-nanotube (CNT)/Polyvinyl alcohol (PVA) composite film-coated thin core fiber (TCF) optics sensor is proposed for monitoring relative humidity (RH). The unique Michelson type fiber interferometer sensor consists a short segment of TCF, the tip of which is melted into a convex shape and coated with a hydroscopic CNT/PVA film. The proposed sensor is linearly responsive to relative humidity (RH) beyond the humidity range 70%RH, with maximum sensitivity of −0.4573 dB/%RH. The advantages of this sensor are its compact size, excellent stability and facile fabrication process.

1. Introduction

Relative humidity (RH) is an important parameter that is closely monitored in agricultural, industrial and chemical fields. Humidity measurement is essential in many applications, such as environmental monitoring, electrical devices and wireless sensor networks, and humidity sensors for high relative humidity measurements have attracted the attention of many groups. Because many devices are easy to be destroyed under high relative humidity, so it is very significant to develop the sensor at the high relative humidity range. Fiber-optic humidity sensors are superior to conventional electrical counterparts in some aspects as the former offers many specific advantages such as compact size and insensitive to electromagnetic interference. Various fiber-optic sensors utilizing various techniques including evanescent wave sensing via etching [1,2], tapering [3,4]; gratings [5–8]; interferometry [9–12] have been reported for monitoring RH. By coating various hydroscopic materials, including organic polymers [4,13], metal oxides [1,6,14] and composite materials [11], the fiber optic sensors are sensitive to RH. However, there exists certain limitations within current fiber optics humidity sensors including the fragileness of etched or tapered fibers, cross-sensitivity of gratings to temperature and strain that limit their feasibility for real world applications. Specialty fibers like photonic crystal fibers have been used in the construction of some of these fiber-optic sensors [9,12], adding to the cost of the fiber sensors.

Due to its large surface area to volume ratio available for the absorption of water/gas molecules, CNTs can be a suitable material for the detection of gases and monitoring of humidity. CNTs are cylindrical carbon molecules with a diameter of few nanometers and possess unique thermal, electrical, mechanical, chemical and optical properties [15]. Already, many researchers have studied the effects of humidity on the electrical conductivity of CNTs. Both CNTs-based composites and pure CNTs have been used for monitoring humidity level. Li et al. fabricated a low humidity sensor based on interdigitated gold electrodes that have been deposited with poly(4-vinylpyridine) that is functionalized with multi-walled carbon nanotubes (MWNTs) [16]. Bradley et al. developed CNTs based field-effect transistors for humidity monitoring [17]. Yoo et al. proposed a resistive-type humidity sensor based on plasma-treated MWCNTs/polyimide composite films, but the sensitivity was just 0.0047/%RH [18]. Adjizian et al. developed pristine CNTs and B- or N-doped CNTs materials for humidity sensing, however the sensor is plagued with long response time [19]. Yu et al. fabricated a composite film consisting poly(ethyleneimine)/MWNT by layer-by-layer assembly, and the sensor showed a fast response in the range of 5%RH to 85%RH [20]. Compared with pure CNTs, CNTs-

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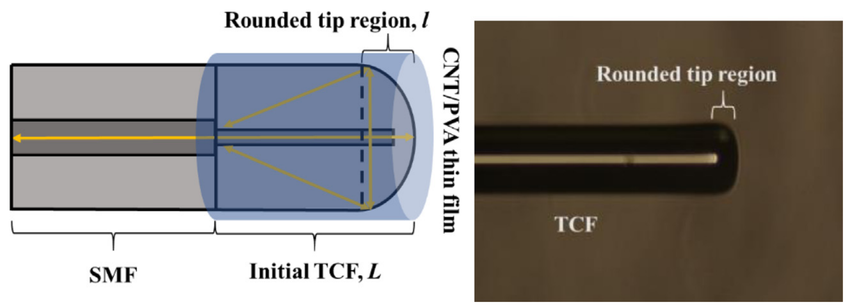

Fig. 1. Schematic diagram of the proposed sensor (*left*); micrograph of the sensor under 10X magnification (*right*).

based composites demonstrate higher sensitivity to humidity. Currently, most researches on RH monitoring devices are focused on achieving high linearity within a wide RH dynamic range. Few studies on the switch-type optical fiber humidity sensors based on intensity modulation are carried out. Such sensors show minimal response below a certain humidity level value and respond sharply thereafter. Such switch-type humidity sensor is important for monitoring and controlling of humidity environments in many fields. Here, we demonstrate the application of CNTs-based composites in an intensity modulated switch-type optical fiber humidity sensor.

A fiber-optic humidity sensor, based on pristine CNT/PVA −coated TCF MI, is fabricated. We present the principles of the TCF based MI sensor and its sensitivity to the external refractive index environment. Next, we coat the MI fiber sensor with CNT/PVA films of varying compositions and characterize the sensor response to RH. The morphologies of the film are also studied and the stability and temperature cross sensitivity of the fiber sensors are empirically determined.

2. Experimental procedures

2.1. Apparatus and materials

PVA (Mw 89,000–98,000, 99+% powder), CNT (98+% carbon basis, O.D. × I.D. × L 10 nm ± 1 nm × 4.5 nm ± 0.5 nm × 3- ∼ 6 μm, multi-walled, powder), Poly (sodium 4-styrenesulfonate) (Mw ∼70,000, powder), Sulfuric acid (95.0-98.0%) and Hydrogen peroxide (30.0%) were obtained from Sigma-Aldrich, Singapore and were used without any further purification. Deionized water was obtained from a MilliQ system. Single mode optical fiber of core diameter 8 μm and cladding diameter 125 μm was bought from Yangtze Optical Fiber and Cable Company Ltd. Thin core fiber, with core diameter of 4 μm and cladding diameter of 125 μm, was purchased from NKT.

The fusion splicer (FSM-100P+) allows customization of arc power, duration and fiber displacement to perform delicate shaping of the fiber tip. The dip coater (KSV NIMA Dip Coater Multi Vessel Small) was used for the reproducible coating of the fiber tip with the CNT/PVA composite. The optical spectrum analyzer (OSA, Yokogawa AQ 6370) was used to acquire the reflection spectral data. The commercial humidity meter (ThermoWorks, DT-3321) with 0.1% RH resolution and 2% RH accuracy was used to calibrate our proposed sensor.

2.2. Preparation of the optical fiber sensor

The sensor was fabricated by splicing one end of a short segment thin core fiber with a single mode lead in/out fiber and melting the other end into a rounded tip (see Fig. 1). In order to obtain a rounded tip, the fusion splicer was set to the splicing parameters as follows: the discharge intensity was set at 7 bit above the standard power, the duration time of discharge was set to 10 s, and the fiber feeding rate was 0.03 μm/ms.

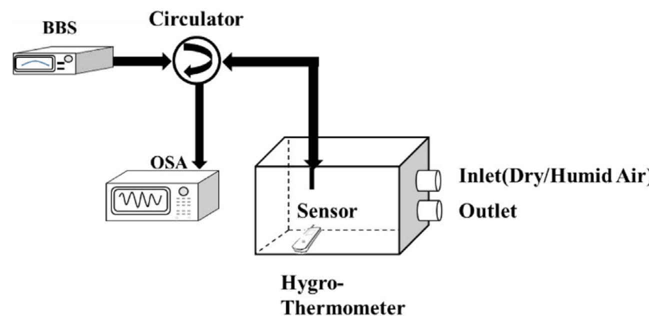

Fig. 2. Experimental set-up for RH measurement.

2.3. Preparation and coating of the composite film

9 wt% PVA solution was prepared by adding PVA granules to deionized water and stirring at 120 °C for 1 h. CNT was dispersed in 1 wt% PSS by ultrasonication. Undispersed CNT was removed by filtration. CNT/PVA blends of varying compositions were prepared by mixing the prepared CNT and PVA solutions in 3:2, 4:1, 1:0, 0:1 vol ratios respectively.

The optical fibers were cleaned with piranha solution consisting sulphuric acid (98%) and hydrogen peroxide (30%) in a 7:3 vol ratio and rinsed thoroughly with deionized water before drying with nitrogen gas. The sensors were dipped into the CNT/PVA blend and withdrawn with a speed of 40 mm/s using the dip coater. After coating, the sensors were left to dry for two days before testing.

2.4. Morphological study of CNT/PVA using FESEM

Samples of the CNT/PVA films were prepared to study their morphologies using the field emission scanning electron microscope (FESEM, JEOL JSM6700) The samples were prepared by spin coating the as prepared CNT/PVA blend on a silicon wafer and allowing them to dry under room conditions for 2 days before further drying in a vacuum oven for a further day. The samples were sputtered with Pt using an auto fine coater (Jeol JFC-1600) to confer electrical conductivity prior to examination using the FESEM.

2.5. Experimental set-up

The set-up comprises a broadband light source (HOYATEK), optical circulator, sensor probe and OSA that are connected as shown in Fig. 2. Light from broadband light source (BBS) is propagated to the sensor probe by the optical fiber circulator. The reflected signal from the sensor is then guided to the OSA for measurement.

For humidity testing, the fiber sensor and hygro-thermometer were placed in a custom-made enclosed chamber, as shown in